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SOME THERMOPHYSICAL PROPERTIES OF BLOOD COMPONENTS AND COOLANTS FOR FROZEN BLOOD SHIPPING CONTAINERS

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SUMMARY

Thermophysical properties of some coolants and blood components at low temperatures were investigated. The results provide sufficient information for the design of the frozen blood shipping container used in the Transportable Blood Transshipment Center (TBTC) program.

Experiments on three different batches of the glycerolized red blood cells demonstrated large differences in the properties of the samples. This finding is an indication of inconsistencies in the procedure for the preparation and glycerolization of red cells. Data obtained from the red blood cell (RBC) sample with the lowest heat capacity exhibited a melt temperature of -24.8 $^{\circ}$ C (-12.6 $^{\circ}$ F), heat of fusion of 65.2 J/g, and specific heat of 1.47 to 2.57 J/g $^{\circ}$ C in the range of -80 to -40 $^{\circ}$ C (-112 to -40 $^{\circ}$ F). The information obtained from this sample should be considered in the design of the frozen blood shipping container.

Several organic compounds suitable to serve as coolants for the shipping container were also studied. Experiments were conducted on 16 coolants; among them, 1-Pentanol, which was approved on its degree of hazard and toxicity by the United States Air Force, was selected as the best choice.

Despite the fact that 1-Pentanol has a low density of 0.862 g/ml at -60 $^{\circ}$ C (-76 $^{\circ}$ F), its melt temperature of -78.5 $^{\circ}$ C (-109.3 $^{\circ}$ F) and total heat absorbing capacity of 145.33 J/g in the range of -80 to -40 $^{\circ}$ C (-112 to -40 $^{\circ}$ F) makes it very attractive as the coolant to be used for the shipping container.

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SOME THERMOPHYSICAL PROPERTIES OF BLOOD COMPONENTS AND COOLANTS SUITABLE FOR FROZEN BLOOD SHIPPING CONTAINERS

1. INTRODUCTION

Since the introduction of modern blood component therapy, red blood cells (RBCs) have largely superseded whole blood in most blood banks. Aside from the fact that RBCs are therapeutically equivalent to whole blood, they offer a distinct but frequently overlooked advantage. Substantially more units of RBCs can be stored and shipped than units of whole blood. The logistical value of being able to ship more blood in a given container without increasing the size or the weight of the container is obvious.

In 1965, the first frozen blood banks were deployed to provide an emergency reserve of universal donor C-positive and O-negative RBCs when liquid blood was not available to meet the needs. The frozen blood banking system provided an alternative to the walking blood donor approach and eliminated a dependency on the presence of healthy men and women to serve as donors at times when demands for blood were high, and in places where blood collection was difficult. However, one of the primary obstacles to frozen blood preservation has been hemolysis—upon thawing, due to osmotic stress on the red cell membrane. The formation of extracellular ice crystals causes dehydration of cells and increases solute concentration in the unfrozen liquid, making the cells less durable (1).

Techniques are now available by which RBCs can be frozen and stored for prolonged periods at very low temperatures. In the most widely used method, red cells are frozen with 40% W/V glycerol in the polyvinyl chloride (PVC) plastic bag and stored at -80 $^{\circ}$ C (-112 $^{\circ}$ F). The addition of glycerol as an agent mitigates cell destruction and reduces the mole fraction of water available for crystallization. After thawing, the glycerol is washed out to prevent osmotic damage to red cells. The cells may then be infused with saline, albumin, or plasma. The RBCs preserved in this way exhibit over 90% survival rate (1).

The Military Blood Program Office (MBPO) is now in the process of implementing a worldwide frozen blood system in which RBCs are stored at -80 °C (-112 °F) and preserved during shipment. This system requires new technologies which will afford the added protection necessary for proper maintenance of the frozen blood. The requirement for devising better ways of solving the logistics problems posed by blood shipment stems from the fact blood is perishable and requires special handling. Permanently attaching a mechanical freezer to a blood shipping container may not be desirable due to increased weight, reduction in usable volume, availability of power, and rejection of heat produced by the condensing unit. Therefore, design criteria for containers for blood must consider weight, size, thermal performance, and mechanical protection of the contents.

A number of commercially available shipping containers were evaluated for general design characteristics and suitability for frozen blood storage (2). A configuration consisting of an insulated box and coolant appeared to be the most feasible for a shipping container. This configuration was then selected as the preliminary choice at the Transportable Blood Transshipment Center (TBTC) Mission Requirements Review Meeting (11/29/88 - 12/1/88).

The problem of maintaining proper temperatures during transit is perhaps the most critical issue. In the past, there has been little interest in the development of the coolants for low temperature shipping containers. Currently, dry ice is being used extensively with frozen products — the amount depending on the type of container and the length of the journey. However, the use of large amounts of dry ice may result in a buildup of carbon dioxide gas in concentrations dangerous to man, unless proper ventilation of the cargo compartment is provided. Moreover, the use of dry ice as a conditioning medium, due to its quantity restrictions on Military Airlift Command (MAC) aircraft, is prohibited.

Since the state of life of frozen RBCs depends on maintaining a temperature between -80 to -40 $^{\rm O}$ C (-112 to -40 $^{\rm O}$ F), the heat transfer capability of the container and coolant in this tempera-

ture range is especially important. Once the overall heat transfer rate of the insulated container is known, the amount of cooling required to maintain RBC in the desired temperature range can be estimated with reasonable accuracy. Thus, a knowledge of the specific heat and latent heat of the glycerolized RBC and favorable coolants are required to predict the heat transfer rate and consequent sizing of the shipper.

1.1 Red Blood Cells Whole blood is divided into about 55 volume percent plasma and about 45 volume percent cells, or "formed elements." The cells consist of about 95% (by number) RBCs, or erythrocytes. White cells comprise another 0.13% by number, and the remainder, about 4.9%, consists of platelets. The plasma portion of the blood is essentially a dilute electrolyte solution containing about 8 weight percent proteins.

The principal functions of the red cells are to transport oxygen and CO₂ throughout the body and buffering the blood so as to regulate pH. The red cell possesses neither a nucleus nor any other detectable internal structure; its shape is of a biconcave disc; its outer membrane is very freely permeable to water, but is relatively rigid so that the cell bursts readily when suspended in a hypotonic medium; the range of enzymes and other proteins present in the cell is more restricted than in nucleated cells. The physical properties of RBC for a normal adult (mean values) are:

| рН | 7.396 |
|--------------------------|--|
| Specific gravity | 1.098 |
| (25/4 °C) | |
| Count - male | 5.4×10^9 /ml whole blood |
| - female | 4.8×10^9 /ml whole blood |
| Life span | 120 days |
| Production rate | 4.5 \times 10 ⁷ /ml whole blood/day |
| Hemoglopin concentration | 0.335 g/ml erythrocyte |

The following thermal properties of packed cells at subzero temperatures have been studied by Rinfret (3).

| emperature | Thermal conductivity | Thermal diffusivity |
|-------------|-------------------------------|--------------------------------------|
| °C (°F) | $W/(Cm^{\circ}C) \times 10^3$ | $\text{Cm}^2/\text{sec} \times 10^3$ |
| -10 (14) | 12.4 | 6.8 |
| -20 (-4) | 13.1 | 8.2 |
| -40 (-40) | 15.1 | 11.0 |
| -60 (-76) | 17.3 | 14.1 |
| -80 (-112) | 19.8 | 17.2 |
| -100 (-148) | 22.6 | 20.4 |
| | | |

Temperature

However, a review of current literature reveals the thermophysical properties of glycerolized RBC have not been investigated. Unique characteristics of RBC cautions the investigators against the extrapolation of data obtained with blood components to RBCs and glycerolized RBCs.

1.2 Glycerol-A Cryoprotective Agent for Red Blood Cells Little or no survival of frozen-thawed RBC samples can be obtained unless a chemical additive is mixed with the cell sample to protect it during the attempted cryopreservation procedure. The modern era of cryobiology began in 1949 with the accidental discovery that glycerol protected spermatozoa during freezing. Since that time, numerous compounds have been tested as potential cryoprotective "antifreeze" (CPA). Today, glycerol is the most common penetrating compound used in practice. The properties of glycerol (4,5,6) are:

| Formula | сн ₂ онснонсн ₂ он |
|------------------|---|
| Characteristics | Pale yellow, syrupy, warm, |
| | sweet taste, hygroscopic |
| Formula weight | 92.1 |
| Melting point | 17.9 °C (64.2 °F) |
| Boiling point | 290 °C (554 °F) at 760 mm. |
| Specific gravity | 1.260 (20/4) |
| Refractive index | 1.1473 |
| Viscosity | 4220 C _p at 2.8 ^o C (37 ^o F) |
| | 1069 C _p at 20 °C (68 °F) |
| | 494 C _p at 26.5 °C (79.7 °F) |
| Heat of fusion | 47.5 g-cal/g |
| Solubility | Infin. in water and alcohol |

There are 2 major cryopreservation procedures applied to RBC in current blood bank practice. The high-glycerol/slow-cooling method uses glycerol concentrations in the range of 40-80% and freezing rates of 1 $^{\rm O}$ C/min (1.8 $^{\rm O}$ F/min) or less. The second major method is the so called "low-glycerol/rapid-cooling technique." This protocol uses glycerol concentrations of approximately 10-20% and cooling rates of 100 $^{\rm O}$ C/min (180 $^{\rm O}$ F/min) (4).

1.3 Coolants Many factors must be considered in selecting a method of cooling for a shipping container. Storage of thermal energy in the form of latent heat of fusion demonstrates attractive features for this purpose. The so-called "latent heat" storage/absorption is accomplished by phase transition caused by heat exchange during which the temperature of an eutectic medium (coolant) remains unchanged. The total amount of thermal energy that can be absorbed by a mole of a certain medium is, to a large extent, determined by the amount of heat involved in phase transitions such as solid to liquid and liquid to gas. The change from one phase to another phase occurs at different temperatures for different materials. Choice of the type of phase change and of materials enables this method of heat absorption to be suitable for frozen blood shipping container over the wide temperature range of -80 to -40 $^{\circ}$ C (-112 to -40 $^{\circ}$ F). The fact that the heat transfer through the coolant is both in the form of sensible and latent heat results in a higher heat absorpting capacity (ratio of heat absorbed to the corresponding temperature rise) as well as smaller size and lower weight per unit of storage.

Determination of the heat capacity of samples in the temperature range of -80 to -40 $^{\rm O}$ C (-112 to -40 $^{\rm O}$ F) is very useful since it represents the total amount of heat that is absorbed by the samples to reach -40 $^{\rm O}$ C (-40 $^{\rm O}$ F) from -80 $^{\rm O}$ C (-112 $^{\rm O}$ F).

The fundamental criteria for an eutectic material, to be used as a coolant in the frozen RBC shipping container, are:

The coolant should have a melting point (mp) in the range of -80 to -40 °C (-112 to -40 °F) to permit latent heat absorption to take place.

- The coolant should have a large heat of fusion. The larger the heat of fusion, the less coolant is required to absorb a given amount of energy.
- The coolant should have high specific heat (heat capacity per unit mass).
- The coolant should be noncombustible, noncorrosive, and nontoxic. In general, the coolant should not be hazardous. Since the possibility of accidental leakage is always present, it is preferable to choose a material with a neutral pH.
- The coolant should have a congruent melting point. The material should melt completely so that the liquid and solid phases are identical in composition. Otherwise, the difference in densities between solid and liquid will cause segregation, which causes changes in the chemical composition of the material.
- The coolant should be chemically and geometrically stable in thermal cycling.
- The coolant should have sufficient mechanical strength to be able to support compression load resulting from the stacking of the RBC packs.
- The coolant should not react with the bag.
- The coolant should be cheap and available.

The technical literature currently available indicates that difficulties have been encountered in obtaining thermophysical properties of a material that will satisfy all the just mentioned criteria.

1.4 Objective The objective of this research is to study and evaluate thermophysical properties of glycerolized RBC and some organic compounds that are suitable for use as coolants in the blood shipping container. In addition to glycerolized RBC,

some properties of whole blood containing citrate-phosphate-dextrose-adenine CPDA-1 and platelets 6% dimethyl sulfoxide DMSO were also evaluated. The specific aims of this project are outlined as follows:

1.4.1 Blood Components

- (a) To evaluate melt temperature or range using dwell time technique to determine both the range of testing and how the measurement was to be carried out.
- (b) To determine the melt temperature by differential scanning calorimeter.
- (c) To determine heat capacity of the solid and liquid phases, including latent heat of fusion by differential scanning calorimeter.
- (d) To determine thermal conductivity in the solid and liquid phases by using an especially designed low temperature cell.
- (e) To determine density of the glycerolized RBC in the solid and liquid phases.

1.4.2 Coolants

- (a) To study and conduct a literature survey of the organic compounds suitable as coolants for the frozen blood shipping container.
- (b) To evaluate melt temperature or range of the selected compounds and their aqueous solutions by dwell time technique to determine both the range of testing and how the measurement was to be carried out.
- (c) To determine the heat capacity of the compounds and their aqueous solutions using an especially designed low-temperature calorimeter.

- (d) To select the aqueous solution of the compounds for further study.
- (e) To determine melt temperature or range of the compounds and their aqueous solutions by differential scanning calorimeter.
- (f) To determine the heat capacity of solid and liquid phases of compounds, including latent heat of fusion by differential scanning calorimeter.
- (g) To determine density of some selected compounds in the solid and liquid phases.

2. METHOD

Standard accepted techniques were chosen for the measurement of each property. However, due to low temperature range of experimentation, some modifications were made.

Of the properties desired, thermal conductivity of the blood components was terminated due to the selection of the type and configuration of the shipping container. Therefore, the data collected during these experiments is incomplete. However, if needed, the process can be continued and completed in the future.

2.1 Samples

- 2.1.1 <u>Blood Components</u> Initially, 3 blood components, glycerolized red blood cells, platelets, and whole blood were tested for melt temperature, latent heat, and heat capacity. Among these blood components, properties of glycerolized RBCs were the most important factor for the design of the shipping container. Thus, similar experimentations were conducted on total of 3 RBC samples. Furthermore, density measurements on 2 more glycerolized samples of RBC, in solid and liquid phases, were also conducted.
- 2.1.2 Coolants A list of 46 organic compounds (see Appendix A) was devised and submitted to the United States Air Force

(USAF) for evaluation of toxicity and degree of hazard. The following 11 compounds from that list seemed to be suitable for frozen blood shipping containers:

- (1) 1 Pentanol

 Heat of fusion:111.5 J/g

 Melting point:-78.9 °C (-110 °F)

 Boiling point:137.3 °C (279.1 °F)
- (2) Propylene-Glycol(60%)
 Heat of fusion: Melting point:-58 OC (-72.4 OF)
- (3) Gamma-Butyrolactone
 Heat of fusion:111.17 J/g
 Melting point:-43.37 °C (-46 °F)
 Boiling point:206 °C (402.8 °F)
- (4) 1,2,4-Trimethylbenzene
 Melting point:-43.8 °C (-46.8 °F)
 Boiling point:169.35 °C (336.8 °F)
- (5) Dimethyl formamide

 Heat of fusion:108.03 J/g

 Melting point:-60.48 °C (-76.8 °F)

 Boiling point:149.56 °C (301.2 °F)
- (6) 1,3,5-Trimethylbenzene
 Heat of fusion:79.08 J/g
 Melting point:-44.7 °C (-48.4 °F)
 Boiling point:164.7 °C (328.4 °F)
- (7) 1,4-Diethylbenzene
 Heat of fusion:78.87 J/g
 Melting point:-42.85 °C (-45.1 °F)
 Boiling point:183.75 °C (362.7 °F)
- (8) Undecane
 Heat of fusion:142.76 J/g

Melting point:-25.6 °C (-14.08 °F)
Boiling point:196 °C (384.8 °F)

- (9) Pyrrole
 Heat of fusion:117.86 J/g
 Meltinς point:-23.41 °C (-10.1 °F)
 Boiling point:130.1 °C (266.1 °F)
- (10) 1,2-Diethylbenzene

 Heat of fusion:108.49 J/g

 Melting point:-31.24 °C (-24.2 °F)

 Boiling point:183.42 °C (362.1 °F)
- (11) Methylchloroacetate

 Heat of fusion:104.01 J/g

 Melting point:-32.12 OC (-25.8 OF)

 Boiling point:129.82 OC (265.6 OF)

The top 4 materials, in addition to the 2 selected by Systems Research Laboratories (SRL), were the final candidates for experimentation. The property measurement experiments were conducted on the following solutions:

- (a) Ethylene glycol and its aqueous solutions of 50%, 52%, 55%, 58%, and 60%.
- (b) Propylene glycol and its aqueous solutions of 50%, 55%, 58%, and 60%.
- (c) 1-Pentanol.
- (d) Gamma-Butyrolactone and its 60% aqueous solution.
- (e) 1,2,4-Trimethylbenzene.
- (f) Saline solution with 25% ethanol.

2.2 Preparation of Samples

2.2.1 <u>Blood Components</u> Glycerolized RBCs, platelets, and whole blood units for this research were provided by Wilford Hall USAF Medical Center (ATC), Lackland AFB, Texas.

The whole blood units were received in PVC bags and contained CPDA-1 anticoagulant. The platelet units were also received in PVC bags. The platelet concentrate was prepared by isolating it from the unit blood by serial differential centrifugation and stored undisturbed at room temperature. Then the platelets were gently manipulated to allow for uniform resuspension.

The red cells were also concentrated by centrifugation of the primary collection bag to obtain a 90 volume %. A glycerol solution of 57 g/100 ml was then added according to glycerolization chart to get the final RBC unit. Each 100 ml of glycerol solution contained 57 g glycerin, United States Pharmacopeia (USP), sodium lactate, 30 mg potassium chloride, USP, buffered with approximately 25 mEq/l of sodium phosphate.

The complete procedure for glycerolization of human RBCs for cryoprotection, prepared by Department of Pathology, Transfusion Branch, Wilford Hall USAF Medical Center, is presented in Appendix B.

- 2.2.2 <u>Coolants</u> The organic compounds, except ethylene glycol, used for experimentation in this study were purchased from Aldrich Chemical. The following physical properties of the samples, and their method of preparation, are provided by the manufacturers:
- (a) Ethylene glycol. This material was provided by Dow Chemical Company, U.S.A. with a product name of Dowtherm SR-1 heat transfer fluid (a trademark of the Dow Chemical Company). A material safety data, sheet as well as the typical physical properties for the product, is included in Appendix C. The following information was supplied by the manufacturer:

INGREDIENTS; (% w/w, unless otherwise noted)

Ethylene glycol >90%
Diethylene glycol < 5%
Dipotassium phosphate < 5%
Water < 5%

PHYSICAL DATA;

Boiling point: 163 °C (325 °F)

VAP press: 2.2 mmHg @ 20 °C (68 °F)

VAP density: >1

Sol. in water: Infinite

Sp. gravity: 1.1295 @ 16 °C (60.8 °F)

Appearance: Pink, liquid

Odor: Pungent odor

FIRE AND EXPLOSION HAZARD DATA:

Flash point: 111 °C (231.8 °F)

Method used: (TCC)

Extinguishing media: Water fog, alcohol foam, CO2,

and dry chemical.

Price \$6.70/qal

Five Dowtherm SR-1 solutions were prepared on a weight percentage basis with the following concentrations: 50%, 52%, 55%, 58%, and 60%. The solution concentrations were arranged by mixing pure SR-1, as supplied from Dow Chemical, with a calculated amount of distilled water. The data was used to determine the ratio of SR-1 to water based on laboratory conditions of 25 °C (77 °F) temperature and ambient pressure of 1 atmosphere; these conditions were closely approximated during the preparation. A density of 1.13 g/ml was used for the pure SR-1, and a density of 0.9973 g/ml was used for water (5).

(b) 1-Pentanol CH₃(CH₂)₄OH 99+% purity F.W. 88.15 boiling point 136-138 °C (276-280 °F) n_D²⁰ 1.4093

d 0.811
Toxic, irritant, combustible liquid

Price \$20.90/4 liters >3000 liters \$ 1.75/liter

(c) Gamma-Butyrolactone 99+% purity

F.W. 86.09

boiling point 204 to 205 °C (399 to 401 °F)

nD 1.4365

d 1.120 irritant, hygroscopic

Price \$42.10/3 kg >3000 kg \$12.00/kg

The aqueous solution of Gamma-Butyrolactone 60% was prepared according to the procedure used for ethylene glycol.

(d) 1,2,4 - Trimethylbenzene $C_6H_3(CH_3)_3$ 98% purity F.W. 120.20 boiling point 168 $^{\circ}C$ (344 $^{\circ}F$) n_D^{20} 1.5040 d 0.889 irritant, combustible liquid

Price \$23.70/kg >3000 kg \$5.00/kg

(e) Sodium chloride A.C.S. reagent Na cl 99+% purity F.W. 58.44
melting point 801 °C (1474 °F)
d 2.165
irritant, hygroscopic

Price \$19.95/2 kg >3000 kg \$ 1.30/kg

(f) Ethyl Alcohol (Ethanol) C_2H_5OD 99.5+% F.W. 47.08

boiling point 78 to 79 $^{\rm O}{\rm C}$ (172 to 174 $^{\rm O}{\rm F}$) ${\rm n_D}^{20}$ 1.3595 d 0.801 flammable liquid, irritant, hygroscopic

Price >3000 g \$ 0.52/g

(g) Propylene Glycol $C_3H_8O_2$ 99.0% F.W. 76.09 boiling point 189 °C (372.2 °F) n_D^{20} 1.4324 d 1.0361

Price >3000 kg \$ 3.80/kg

The aqueous solutions of propylene glycol 50%, 55%,
58%, and 60% were prepared according to the procedure
used for ethylene glycol.

2.3 Experimental Procedure

2.3.1 Cryogenic System To achieve cryogenic temperatures, a liquid nitrogen system was devised. Many hazards are related to the use of liquid nitrogen, which were considered in the overall design of the system. For example, liquid nitrogen, under certain circumstances, may condense oxygen from the atmosphere, causing oxygen enrichment or entrapment in unsuspected Extremely cold metal surfaces may also condense oxygen from atmosphere. The formation of oxygen will then result in a The large expansion ratio from the liquid flammability hazard. to the gas (696.5 to 1) for nitrogen provides a source for build up of high pressure. The body contact at low temperatures is another concern which is capable of causing burns like thermal burns. Long exposure will also result in the embrittlement of the exposed body member. Precautions were considered in the design and operation of the liquid nitrogen system with full protection of the operators.

The cryogenic system consists of a liquid nitrogen (LN_2) tank, a dry nitrogen gas (N_2) tank, and a vacuum-insulated cylinder (dewar). Initially, the dewar was filled with liquid nitrogen

at -194 °C (-317.2 °F) via a flexible transfer hose. The level of liquid was detected and controlled by a level controller and a solenoid valve. Dry nitrogen gas at room temperature was then introduced to the dewar cylinder, where the appropriate heat exchange takes place. A copper-constantan thermocouple was mounted in the cylinder to monitor temperature. The flow of nitrogen was controlled by a DM20 servo-valve receiving signals from the HP 3497A data acquisition system and IBM Model 60 micro-computer (Fig. 1).

- 2.3.2 <u>Freezing Point Inspection</u> To evaluate the freezing temperature and condition of the aqueous solutions below -30 °C (-22 °F) a test was conducted. In this experiment the solutions were stored in 250 ml Pyrex, long neck, laboratory tubes. The tubes were then placed in a refrigerated, temperature controlled bath, which gradually reduced the temperature of each of the samples. The temperature was reduced from 25 to -32 °C (77 to 25.6 °F) over a period of about 4 h. The samples were examined approximately every 30 min. during the chilling process. Thermocouples were placed in the sample containers and temperature bath to verify the temperature regulation within the bath.
- 2.3.3 Heat Capacity and Latent Heat A differential scanning calorimeter (DSC) was leased to evaluate the heat capacity and latent heat of fusion of the samples at low temperatures. However, due to high operating cost and expenses of the DSC, a less accurate non-adiabatic calorimeter was used to determine the aqueous solutions of the coolants for final evaluation.
- 2.3.3.1 <u>Non-adiabatic Calorimeter</u> To evaluate the heat capacity of the organic compounds and their aqueous solutions, a non-adiabatic calorimeter was used. This calorimeter, which was designed and fabricated at the University of Texas at San Antonio, incorporates a relaxation method and is capable of measuring heat capacity of liquids at very low temperatures.

This apparatus can be connected to the existing cryogenic system for low temperature measurements. However, the accuracy of this system is much lower than the DSC. Therefore, the non-adiabatic calorimeter was used, initially, to evaluate heat capacity of

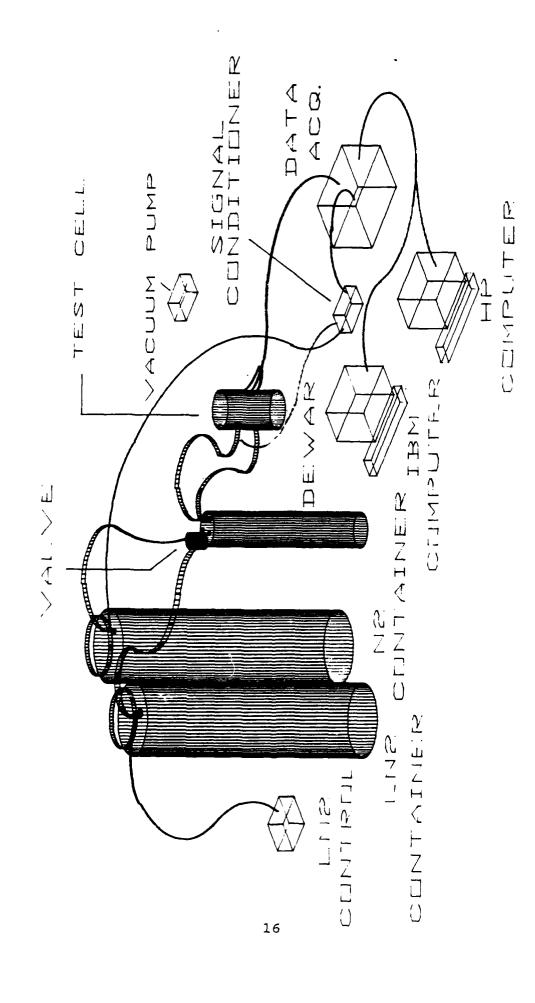


Figure 1. The Cryogenic System

different materials and their aqueous solutions. The solutions that presented good heating capacity were selected for further investigation with DSC.

The apparatus consists of a holder for the liquid sample which was attached to a heat sink via a weak link. A stainless steel cover was placed over the sample holder and weak link, which was evacuated during heating cycle (Fig. 2). The system was initially cooled to the required temperature, T_0 , by circulating nitrogen gas from the cryogenic system through both upper and lower chambers. When equilibrium was achieved, the flow of nitrogen gas to the upper chamber was stopped, while gas continued to flow through the lower chamber to maintain the heat sink at T_0 . During heating and cooling cycles, the sample was surrounded by a vacuum. In addition, the calorimeter was insulated by evacuation of the space under the cover. Foil-covered Fiberglas insulation surrounded the entire system, and the system was then placed in a Styrofoam casing.

During the heating cycle, the one-dimensional energy equation, from the First Law of Thermodynamics, was:

$$mC_{p} [dT/dt]_{h} = Q_{h} - Q_{l} + Q_{s}$$
 (1)

where $[\mathrm{dT/dt}]_h$ was the rate of temperature change of the sample, Q_h was heat added by the heater, Q_1 was the heat lost through the weak link, Q_S was the total stray heat, and m was the mass of the sample. When the sample had reached the required temperature, the heater was turned off and the sample was allowed to cool.

During the cooling cycle, the one-dimensional energy equation used was:

$$mC_{p} [dT/dt]_{c} = -Q_{1} + Q_{s}$$
 (2)

where $\left[\mathrm{d}T/\mathrm{d}t \right]_{\mathrm{C}}$ was the rate of temperature change for the cooling process.

The sample temperature was then plotted as a function of time to obtain cooling as well as heating curves. The \mathbf{Q}_1 was assumed to

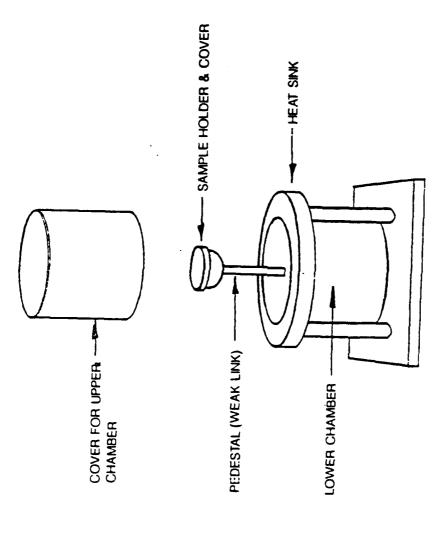


Figure 2. Diagram of Calorimeter

be the same for both the heating and the cooling process and $Q_{\rm S}$ was nearly the same for both processes. Then the equations for heating and cooling were solved simultaneously to obtain:

$$mc_{p} = \frac{Q_{h}}{[(dT)/(dt)_{h} - (dT)/(dt)_{c}]}$$
(3)

Thus, C_p was determined at a given temperature by finding the difference between the slopes of the heating and cooling curves at this temperature, using the following equation:

$$c_{p} = \frac{Q_{h}}{m[(\Delta T)/(\Delta t)_{h} - (\Delta T)/(\Delta t)_{c}]}$$
(4)

Where \textbf{Q}_h and m were measured and $\left[\Delta T/\Delta t\right]_h$ and $\left[\Delta T/\Delta t\right]_C$ were determined from the heating and cooling curves.

It was imperative that heat be added slowly, and the heat sink be kept at $T_{\rm O}$ to maintain the sample in thermal equilibrium and simulate a reversible process.

A model 216, HP 9000 series computer was used with an HP-3497A data acquisition unit. An IBM Model 60 microcomputer was also used to analyze the data.

A program in HP-Basic was written to monitor the temperatures of the sample and the heat sink using copper-constantan thermocouples. The temperature readings, time, and voltage of the heater were displayed on the screen and recorded on the hard disk. The program also controlled the flow of nitrogen gas via the DM20 servo-valve. The light bulb used to heat the sample was turned on by the program when equilibrium of the system was achieved and turned off when the sample reached a predetermined temperature.

The system was calibrated using a standard with a known specific heat. The heat capacity of the empty sample holder and the standard were measured and a calibration curve for the desired temperature range was obtained. 2.3.3.2 <u>Differential scanning calorimeter</u> A Dupont 910 DSC, and a Dupont 1090B TA thermal analyzer, along with a Dupont 1091 Disk Memory, were used for accurate heat flow measurements at very low temperatures. This system measures temperatures and heat flow associated with material transitions, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) problems.

The system is made up of a cell base module and a DSC cell (Fig.3). The cell base is an operating base for the DSC cell. Connected by cable to the control console, it transmits heater voltages and thermocouple signals between the console and the cell. Output from the thermocouples is controlled and amplified by circuitry in the cell base. All system controls are located on the front of the cell base. Input ports for vacuum, cooling, and purge gases; connectors; and calibration controls are located at the rear of the cell base.

The system's measuring unit is a plug-in DSC cell, which is used to measure differential heat flow. The unit is equipped with a holder for LN_2 . In the cell, the sample and an empty reference pan sit on raised platforms which are on a constant disc. Heat is transferred through the disc to the sample and reference. Differential heat flow is monitored by thermocouples located beneath the disc.

The specifications of the calorimeter are:

CELL BASE MODULE

Dimensions: L 30 cm (11.9 in.)

W 45 cm (18.1 in.)

H 14 cm (5.7 in.)

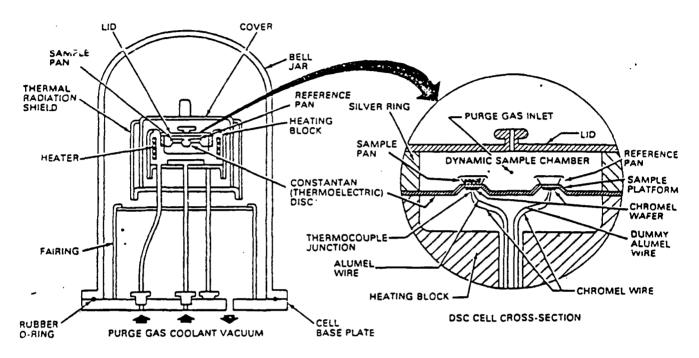
Weight (approx): 8.2 kg (18 lb)

Power: ± 5.6 VDC, and ± 15 VDC

supplied by programmer 115 VAC heater voltage

DSC CELL

Dimensions: D 13 cm (5.2 in.)



Cross Section, DSC Cell

Figure 3. Differential Scanning Calorimeter Cell

H 19 cm (7.3 in.)

Weight (approx): 2.3 kg(5 lb)

Temperature range: Room temperature to 725 OC

(1337 °F), inert atm above

600 °C (1112 °F) as

supplied, to -180 °C (-292 °F) with subambient

accessories

Cooling rate: Dependent of accessory used

and temp range

Sample size: 0.5 to 100 mg (nominal)
Sample volume: 10 mm³ in hermetic pans

Sample pans: Various open or hermetically

sealed

Atmosphere: Atmospheric to 266 Pa (2

torr): preheated dynamic gas
purge (in excess of

 $100 \text{ cm}^3/\text{min}$

Cell Volume: 2 cm³

Temperature repeatability: ±1 °C (1.8 °F)

Differential thermocouples: Chromel-constantan

Sample thermocouple: Chromel-alumel
Control thermocouple Platinel II

Calorimetric precision: Normal - 6 W/cm (0.003

mcal s⁻¹/inch)

Calibrated - 10 W/cm(0.005

mcal's⁻¹/inch)

Temperature calibration: Prior to experimentation the following temperature calibration procedure was used to adjust temperature readings of the recorder and programmer to a standard with a known melting point.

The sample thermocouple was calibrated using a 10-mg indium sample and an aluminum sample pan. Using an empty sample pan with a lid as the reference, the indium sample was scanned. Difference in transition temperature between actual onset and standard was then corrected and adjusted on the back of the cell base.

Calibration coefficient E: The cell base electronics provided a linear calibration coefficient (E) curve. This coefficient was constant, nominally 200 W/mV with the MODE switch at DSC.

Indium was used for this calibration, because its heat of fusion is well known. To determine E:

- 10 mg indium sample was crimped into an aluminum sample
- An empty sample pan with a lid for reference was used and the indium sample was programmed through its melt.
- The E value at the transition peak from the area under the curve using the known heat of fusion of indium (28.4 J/g [6.79 mcal/mg]) was calculated.

Using a material with a known heat capacity at a given temperature can determine the calibration coefficient at that temperature with the following equation:

$$E = \frac{C_p H_r m}{60 \Delta q_s \Delta Y}$$
 (5)

where C_p = Heat capacity at the temperature of interest in $J/q^{O}C$

H_r = Heating rate in ^OC/min

m = Sample mass in mg

 $\Delta q_s = Y$ -axis RANGE setting in mV/cm

ΔY = Difference in Y-axis deflection between sample and blank traces at temperature of interest in cm

E = Cell calibration coefficient in mW/mV

If E, as determined in Equation (5), was not 200 mW/mV, the value was used as is.

Calorimetric measurement: The programming was started well below -80 $^{\circ}$ C (-112 $^{\circ}$ F). This measurement allowed the heating rate to stabilize at the set rate and permitted the sample and reference platforms to equilibrate.

Since the heating run started below -80 $^{\circ}$ C (-112 $^{\circ}$ F), the sample was loaded at ambient temperature then cooled to below -80 $^{\circ}$ C (-112 $^{\circ}$ F). The external cooling was provided by continuously filling the cooling chamber, located on the top of the DSC cell, with liquid nitrogen.

The peak area was used to calculate heat of fusion (H) by substitution into the equation:

$$H = (60BE\Delta q_s) A/m$$
 (6)

where A = Peak area in cm^2

m = Sample mass in .mg

B = TIME BASE setting in min/cm

E = Cell calibration coefficient at the temperature of experiment in mW/mV

 $\Delta q_s = Y$ -axis RANGE setting in mV/cm

H = Heat of fusion in J/g

Note that the quantity (60BE Δq_s) is always constant for any given set of instrument conditions. The quantity can be used to convert area directly into heats of reaction.

Determining heat capacity: To determine the heat capacity of the test specimen, the heat flow difference between sample and reference under blank and sample conditions were compared as follows:

- Empty sample and reference pans were loaded.
- The starting and limit temperatures, over the temperature range desired, were set.
- The system was allowed to equilibrate for 5 min.
- The empty pans were programmed at the rate of 10 $^{
 m O}$ C/min. (18 $^{
 m O}$ F) Deflection from the initial equilibrium point was up or down, depending on the heat capacity difference between the pans.
- This procedure was repeated under identical conditions with a weighed sample in the sample pan.
- Heat capacity was calculated by measuring the difference in Y-axis displacement (calorimetric differential) between the sample and blank curves at any desired temperature. See figures in results section.
- The difference was then substituted into the following equation:

$$C_{p} = [60E\Delta q_{s}/H_{r}] \quad \Delta Y/m \tag{7}$$

where E = Cell calibration coefficient at the temperature of interest in mW/mV

 Δq_s = Y-axis RANGE setting in mV/cm

 H_r = Heating rate in OC/min

ΔY = Difference in Y-axis deflection between sample and blank curves at temperature of interest in cm

m = Sample mass in mg

 C_p = Heat capacity in J/g^OC

Note that the quantity (60E $\Delta q_s/H_r$) is constant under a given set of experimental conditions. This equation converts the Y measurement directly into units of heat capacity in J/g^0C . For highest accuracy, the value of this constant (as an entity) was determined by running a sapphire (Al₂O₃) standard material, which has known specific heat under identical conditions, as the unknown sample. Then values of Y, m, and C_p were substituted for the standard into equation (7) at the temperature of interest.

2.3.4 Thermal Conductivity The thermal conductivity, from a physical point of view, demonstrates how well a material transports heat by conduction. The major requirement in the design of a thermal conductivity cell is that the total heat supplied should be used to establish the observed temperature distribution within the specimen. Therefore, for accurate measurements, undesired heat conduction to or from the test section and thermal radiation must be minimized. Liquids present additional difficulties, such as convection currents and trapped air within the liquid layer, which may cause errors in measurements. Another factor having a significant effect on the design of the cell is that the fluids have to be contained prior to experimentation.

The comparative method uses 2 reference standards which are placed on either side of the sample material. The sample and reference standards are, in turn, held between a heat sink and a heat source. To avoid radial heat loss from the sample stack, the surrounding temperature is controlled to match the profile in the test stack to produce one-dimensional heat flow down to the test section. To accomplish this task, a guard tube with independent heaters, located adjacent to each interface of the test stack, is placed around the test section. The heaters in the guard tube are adjusted to match its temperature profile with the test section.

Under steady-state conditions, the one-dimensional heat flow through the sample and the 2 reference standards are equal.

$$Q/A_{Sample} = Q/A_{Top}$$
 Standard $= Q/A_{Bottom}$ Standard (8)

$$k_s \Delta T_s / \Delta X_s = k_{TS} \Delta T_{TS} / \Delta X_{TS} = k_{BS} \Delta T_{BS} / \Delta X_{BS}$$
 (9)

Thus the conductivity of the sample is based on the Top Standard, the Bottom Standard, or an average between both the Top and the Bottom Standards.

$$k_{s} = 1/2 \Delta X_{s} / \Delta T_{s} (k_{TS} \Delta T_{TS} / \Delta X_{TS} + k_{BS} \Delta T_{BS} / \Delta X_{BS})$$
 (10)

The appropriateness of this method was determined by such factors as the temperature range of interest, the range of thermal conductivity values, the physical nature and structural integrity of the material, the geometry or boundary conditions of the available samples, the required accuracy of the data, the speed of measurement, and the effort required.

The schematic of the test cell, which uses the just mentioned method, is shown in Figure 4. The test cell used 2 aluminum reference standards, 4 in. long x 4 in. wide x 2 in. thick, placed on either side of a sample chamber. The chamber was 7.6 cm (3 in.) in diameter and 0.317 cm (0.125 in.) thick. The test cell was cooled from below via a cooling chamber (heat sink) and heated above via a heating chamber (heat source). Six thermocouples were embedded in reference standards, and 6 more thermocouples were placed flush with the surface of the sample chamber. In addition, 1 thermocouple monitored the dewar cylinder temperature, and 1 recorded the room temperature. All thermocouples were copper-constantan type with 30.5 cm (12 in.) probe.

The thermal conductivity cell was placed horizontally so heat flux was in the direction of the gravitational field. Since heat flow through the liquid layer was heated from the top, it minimized the contribution from natural convection. Two vents placed at the sides of the device were used to add liquid to the test chamber and to ensure that the cell was fully filled during the measurement.

Methods used here to measure thermal conductivity do not differ in principle from those used at higher temperatures. However, heat capacities of a system at low temperatures are small and a little heat may produce a large rise in temperature. To minimize this undesired heat transfer, 0.95 cm (3/8 in.) thick Teflon was

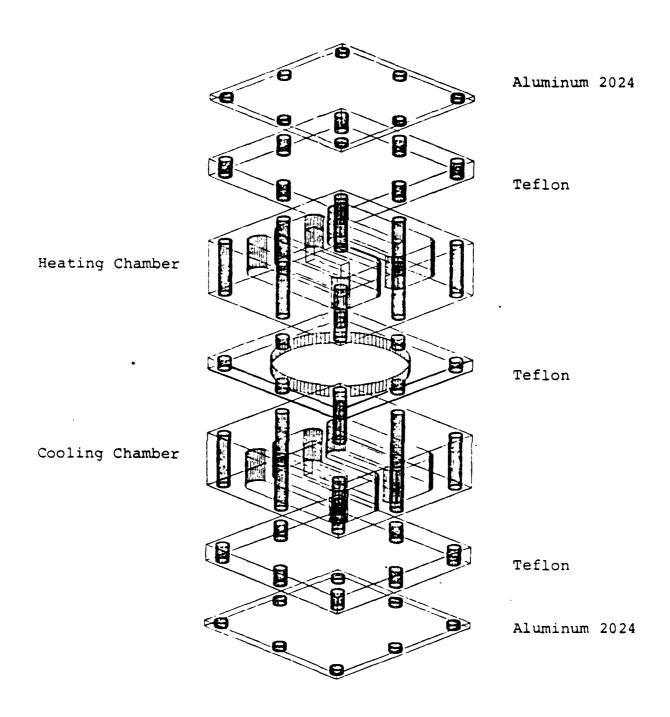


Figure 4. Schematic of Test Cell

used, which covered the exterior of the test cell. The unit was then placed in a rectangular shaped Styrofoam box.

The data acquisition and reduction system consisted of an IBM model 60 microcomputer, HP model 216 computer, HP model 3497A data acquisition, PS circuit power supply, HP current source, 555 timer, HP model 7550A plotter, and IBM printer. The experiment was continuously monitored by the computer controlling the servo-valve, heater, and thermocouples. The initial temperature difference between the heat source and heat sink, in addition to starting and ending temperatures, were input into the computer.

Initial steps were taken to ensure the test cavity in the cryogenic cell was clean and dry before any sample material was introduced for testing. The procedure involved flushing out the cavity with distilled water for about 10 min. The cavity was then dried, using compressed air, for about 20 min. nally located ports, leading to the test cavity, provided access to the cell's interior for the cleaning procedure. Port #1 lead to a bottom opening in the test cavity, whereas, port #2 lead to Twenty five cm (10 in.) of Tygon^R tubing was attached to each of the external port fittings to facilitate filling of the test cavity. Liquid was introduced into the test cavity through the Tygon^R tubing at port #1, using a standard 10 cm³ laboratory syringe (without needle). Air, as well as excess sample material, escaped from the test cavity through port #2 during the filling process. The cavity was considered full when no additional air bubbles escaped through port #2 while filling the cavity.

Prior to data collection, the cell was placed inside a polystyrene housing for increased insulation and thermal stability. The cell resided inside the housing throughout the duration of the test. Thermocouple probes and nitrogen gas pipes were inserted into the test cell through predrilled holes in the polystyrene housing.

The previously described cryogenic cooling system was used to reduce and maintain the temperature of the sample material inside the test cavity. Liquid nitrogen, at -194 $^{\circ}$ C (-312 $^{\circ}$ F), was

piped into a dewar cylinder (insulated holding cylinder). The level of LN_2 inside the cylinder was maintained by the computer. Dry nitrogen gas was then piped through copper coils which were immersed in the liquid nitrogen. The temperature of the dry nitrogen gas leaving the copper coils was adjusted by the computer controlled servo-valve.

A duration of 6 to 12 h was required to attain a steady-state condition for a single data point, depending on the initial temperature distribution in the cell and across the liquid layer.

2.3.5 <u>Density Measuring Unit</u> A Haake A82 cold bath, Ohaus GT 4100 electric top loading balance and copper-constantan thermocouples were used to measure volume changes of the samples. Precision bored Pyrex tubes, which have negligible thermal expansion coefficient compared to the samples, were filled and placed in the cold bath. The volume changes of the samples were then accurately measured at each interval after thermal equilibrium was established. Dry ice was used to assist the bath at temperatures below -20 $^{\circ}$ C (-4 $^{\circ}$ F). The samples were weighed before and after experimentation.

3. RESULTS AND DISCUSSION

3.1. Melting Temperature and Latent Heat The selected samples were cooled down well below -80 °C (-112 °F) and then heated at a set rate. The melt point of the materials was then determined from the intersection of lines drawn through the portions of the temperature curve immediately below and above the peak. Tables 1-4 present the data for the melting onset temperature, melting point, and latent heat of fusion of blood components and coolants. Some of the coolants did not exhibit a visible melt region for the temperature range of experimentation. In addition, some of the coolants exhibited heat evolution (exothermic process) during heating.

Small degrees of supercooling was noticed during the freezing process of glycerolized RBC and some aqueous solutions of ethylene glycol and propylene glycol.

- 3.2 <u>Specific Heat</u> The results of heat input as a function of the sample temperature are summarized in Tables 5-11 and Figures 5-26. The present specific heat results extend the temperature range for these materials over the complete range of use.
- 3.2.1 <u>Blood Components</u> The experimental specific heat results in the solid and liquid phase for glycerolized RBCs are presented in Table 12 and Figures 27-29. Furthermore, Figures 30 and 31 exhibit the specific heats of the 3 samples of glycerolized RBCs. The results for each RBC indicate that the glycerolization procedure is not consistent and the quantity of RBC and glycerol in the solution varies from one batch to another. This difference is greatest between temperatures of -70 to -60 $^{\circ}$ C (-94 to -76 $^{\circ}$ F). For example, specific heat of RBC-A at -66 $^{\circ}$ C (-86.8 $^{\circ}$ F) is approximately 25% higher than that of RBC-C. The results of the specific heat near melting point actually present the effective specific heat values, since they include the effect of latent heat of fusion.

The experimental results of specific heat for platelet, whole blood, and one sample glycerolized RBC are presented in Figure 32.

3.2.2 <u>Coolants</u> The experimental results for coolants are summarized in Tables 13-18 and Figures 33-48. Again, the results of the specific heat near melting point actually present the effective specific values. Most of the aqueous solution did not exhibit a true phase transition. In addition, some of the aqueous solutions showed exothermic problems during heating.

Among all Dowtherm SR1 aqueous solutions, SR1-50% exhibited the best heating capacity. However, between temperatures of -90 and -70 $^{\circ}$ C (-130 and -94 $^{\circ}$ F), an exothermic problem was noticed.

Among all propylene glycol solutions, 60% solution exhibited the best heating capacity. None of the solutions showed signs of true phase transition within the experiment's temperature range.

Specific heat of Pentanol was approximately 1.55 $\rm J/g^OC$ between temperatures of -60 and -40 $\rm ^OC$ (-76 and -40 $\rm ^OF$), with phase transition occurring between -84 and -63 $\rm ^OC$ (-119.2 and -81.4 $\rm ^OF$).

Both samples of Gamma-Butyrolactone had a specific heat value below 1.3 J/g $^{\circ}$ C, for temperatures below -54 $^{\circ}$ C (-65.2 $^{\circ}$ F). However, they presented a phase transition near -40 $^{\circ}$ C (-40 $^{\circ}$ F).

Experimental results of specific heat for platelet, whole blood, and one sample glycerolized RBC are presented in Figure 32.

Specific heat of Trimethylbenzene between temperatures of -80 $^{\rm O}{\rm C}$ (-112 $^{\rm O}{\rm F}$) and -58 $^{\rm O}{\rm C}$ (-72.4 $^{\rm O}{\rm F}$) was below 1.5 $J/g^{\rm O}{\rm C}$. Figure 49 also presents the heat capacity of 1-Pentanol and 1,2,4-Trimethylbenzene.

Saline solution with 25% Ethanol exhibited a phase transition at approximately -58 $^{\circ}$ C (-72.4 $^{\circ}$ F), with an average specific heat of 1.54 J/g° C in solid phase and an average specific heat of over 2.1 J/g° C in liquid phase.

Figure 50 exhibits the heat capacity of 3 aqueous solutions of ethylene glycol (SR-1) 50%, propylene glycol 50%, and saline solution with 25% ethanol.

3.3 <u>Density</u> Experiments on the density of 2 samples of RBCs during freezing and thawing were conducted, and the results are presented in Tables 19 and 20. An inspection of these tables exhibit small density variations in the temperature range of the experiment. The volume of samples increases with decrease in temperature.

The results of density as a function of temperature for Dowtherm SR1-50%, 1-Pentanol and 1,2,4-Trimethylbenzene are presented in Tables 21 and 22.

TABLE 1. MELTING POINT AND LATENT HEAT OF FUSION OF BLOOD COMPONENTS

| | RBC-A | RBC-B | RBC-C | Whole blood | Platelet |
|-----------------------------------|-------|-------|-------|-------------|----------|
| Melting onset temperature [OC] | -50.0 | -60.0 | -52.0 | 22.0 | -31.0 |
| Melting point [OC] | -21.9 | -26.3 | -24.8 | -0.3 | -0.8 |
| Heat of fusion [J/g] | 95.7 | 78.6 | 65.2 | 315.0 | 276.0 |

TABLE 2. MELTING POINT AND LATENT HEAT OF FUSION OF AQUEOUS SOLUTIONS OF ETHYLENE GLYCOL (DOWTHERM SR-1)

| | 50% | 52% | 55% | 58% | 60% | 100% |
|-----------------------------------|-------|-------|-------|-----|-----|--------|
| Melting onset temperature [OC] | -56.0 | -60.0 | - | - | - | -49.0 |
| Melting point [OC] | -48.2 | -55.9 | -51.0 | - | - | -26.8 |
| Heat of fusion [J/g] | 26.4 | 2.2 | 2.4* | - | - | 140.0* |

^{*} Exothermic process occurred during heating of the sample in the desired temperature range.

No visible melt region.

TABLE 3. MELTING POINT AND LATENT HEAT OF FUSION OF AQUEOUS SOLUTIONS OF PROPYLENE GLYCOL

| | 50% | 55% | 58% | 60% | 100% |
|-----------------------------------|-------|------------|-----|-----|------|
| Melting onset temperature [OC] | -49.0 | . - | - | - | - |
| Melting point [OC] | -44.6 | -55.9 | - | - | - |
| Heat of fusion [J/g] | 4.0 | - | - | - | - |

⁻ No visible melt region.

TABLE 4. MELTING POINT AND LATENT HEAT OF FUSION OF SOME COOLANTS

| | | | | | |
|-----------------------------------|-------------|---------|-------------------|-------|--------------------|
| | GB-60%ª | GB-100% | PENT ^b | TRIC | $_{	t ETH}^{	t d}$ |
| | | | | | |
| Melting onset temperature [°C] | -51.0 | -61.0 | -84.0 | -70.0 | -72.0 |
| Melting point [OC] | -46.2 | -46.4 | -78.5 | -50.3 | -60.0 |
| Heat of fusion [J/g] | 59.1* | 109.0 | 115.0 | 80.8 | 4.0* |

^{*}Exothermic process occurred during heating of the sample in the desired temperature range.

a_{Gamma-Butyrolactone}

b₁-Pentanol

C_{1,2,4}-Trimethylbenzene

dSaline Solution with 25% Ethanol

TABLE 5. HEAT FLOW OF GLYCEROLIZED RED BLOOD CELL

| Temperature •C | RBC-A (mW) | RBC-B (mW) | RBC-C (mW) |
|---|--|--|--|
| -90.00 -88.00 -86.00 -84.00 -82.00 -80.00 -78.00 -76.00 -74.00 -72.00 -70.00 -68.00 -66.00 -64.00 -62.00 -60.00 -58.00 -56.00 -58.00 -54.00 -52.00 -50.00 -40.00 -38.00 -34.00 -34.00 -32.00 -30.00 -34.00 -32.00 -30.00 -16.00 -14.00 -14.00 | II . | | |
| -12.00 -10.00 -8.00 -6.00 -4.00 -2.00 0.00 | -18.68 -17.59 -11.25 -8.50 -7.66 -7.27 -6.97 | -36.59 -35.76 -29.75 -24.02 -20.22 -18.33 -17.64 | -26.78 -23.47 -17.74 -14.48 -13.34 -13.04 -13.15 |

TABLE 5. (CONT.)

| Temperature | RBC-A | RBC-B | RBC-C |
|--|--|--|--|
| •C | (mW) | (mW) | (mW) |
| 2.00 | -6.86 | -17.54 | -13.38 |
| 4.00 | -6.85 | -17.83 | -13.80 |
| 6.00 | -6.86 | -18.20 | -14.15 |
| 8.00 | -6.90 | -18.78 | -14.40 |
| 10.00 | -6.93 | -19.37 | -14.91 |
| 12.00 | -6.93 | -20.02 | -15.67 |
| 14.00 | -6.96 | -20.71 | -16.05 |
| 16.00 | -7.14 | -21.56 | -16.84 |
| 18.00 | -7.45 | -22.39 | -17.96 |
| 20.00 | -7.71 | -23.37 | -19.11 |
| 22.00 24.00 26.00 28.00 30.00 32.00 34.00 36.00 38.00 40.00 | -7.87 -7.99 -8.16 -8.37 -8.54 -8.72 -8.82 -8.93 -9.06 -9.23 | -24.56 -25.83 -27.20 -28.52 -30.43 | -20.14 -20.62 -21.94 -23.64 -25.44 -27.01 -28.45 -30.38 -32.53 |

TABLE 6. HEAT FLOW OF DOWTHERM SR1

| Temperature • C | SR1-50% (mW) | SR1-52% (mW) | SR1-55% (mW) | SR1-58% | SR1~60% | 1 |
|------------------|------------------|-------------------|-------------------|------------------|------------------|-------------------|
| | (mw) | (ши) | (ши) | (mW) | (mW) | (WM) |
| -90.00 | -10.05 | -8.29 | -8.45 | -12.64 | -13.57 | -9.82 |
| -88.00 | -8.89 | -8.34 | -8.40 | -12.72 | -13.77 | -9.66 |
| -86.00 | -5.62 | -8.30 | -8.38 | -12.68 | -13.53 | -9.26 |
| -84.00 | 1.57 | -8.25 | -8.36 | -12.62 | -13.28 | -9.22 |
| -82.00 | 11.57 | -8.22 | -8.23 | -12.72 | -13.46 | -9.30 |
| -80.00 | 11.55 | -8.21 | -8.22 | -12.64 | -13.23 | -9.36 |
| -78.00 | -2.75 | -8.22 | -8.14 | -12.61 | -13.24 | -9.25 |
| -76.00 | -6.97 | -8.17 | -8.07 | -12.47 | -13.26 | -8.66 |
| -74.00 | -8.71 | -8.10 | -8.02 | -12.51 | -13.10 | - 7.98 |
| -72.00 | -9.62 | -8.06 | - 7.95 | -12.54 | -13.15 | -7.21 |
| -70.00 | -10.27 | ~8.06 | -7.71 | -12.37 | -13.15 | -5.48 |
| -68.00 | -10.79 | -8.12 | - 7.46 | -12.41 | -13.04 | -2.28 |
| -66.00 | -11.24 | -8.12 | - 6.76 | -12.40 | -12.77 | 2.91 |
| -64.00 | -11.53 | -8.22 | -5.94 | -12.55 | -12.93 | 10.10 |
| -62.00 | -12.17 | 6د.8- | -5.06 | -12.63 | -12.92 | 20.13 |
| -60.00 | -12.34 | -8.38 | -4.29 | -12.76 | -12.95 | 32.58 |
| -58.00 | -12.26 | -8.39 | -4.24 | -12.81 | -12.98 | 47.00 |
| -56.00 | -12.32 | -8.56 | -5.06 | -12.57 | -12.93 | 61.73 |
| -54.00 | -12.48 | -9.04 | -6.64 | -12.55 | -12.86 | 80.19 |
| -52.00 | -12.89 | -9.57 | -8.30 | -12.59 | -12.87 | 99.06 |
| -50.00 -48.00 | -13.28 -13.78 | - 9.60 | - 9.80 | -12.64 | -12.86 | -7.67 |
| -48.00 -46.00 | -13.78 | - 9.45 | -10.92 | -12.85 | -12.88 | -8.33 |
| -44.00 | -14.94 | -9.25 -9.01 | -11.76 | -12.90 | -12.93 | -8.82 |
| -42.00 | -15.65 | -8.82 | -12.43 | -12.95 | -12.76 | -9.39 |
| -40.00 | -16.28 | -8.78 | -12.87 -12.65 | -13.07 -13.00 | -12.97 | -9.82 |
| -38.00 | -17.12 | -8.75 | -11.17 | -13.09 -13.23 | -13.39 | -10.74 |
| -36.00 | -17.68 | -8.86 | -10.01 | -13.23 -13.38 | -13.10 -13.19 | -11.78 |
| -34.00 | -18.71 | -8.94 | -9.49 | -13.36 -13.29 | -13.19 -13.26 | -12.94 |
| -32.00 | -18.75 | -9.06 | -9.18 | -13.33 | -13.26 -13.47 | -14.16 -15.70 |
| -30.00 | -16.00 | -9.20 | -9.10 | -13.35 | -13.47 -13.51 | -17.76 |
| -28.00 | -13.67 | -9.18 | -9.12 | -13.38 | -13.65 | -19.77 |
| -26.00 | -12.54 | -9.34 | -9.28 | -13.60 | -13.91 | -22.04 |
| -24.00 | -12.05 | -9.49 | -9.39 | -13.65 | -14.08 | -25.21 |
| -22.00 | -11.87 | -9.56 | -9.48 | -13.70 | -14.27 | -28.97 |
| -20.00 | -11.76 | -9.64 | -9.58 | -14.03 | | -33.08 |
| -18.00 | -11.78 | -9.85 | -9.60 | -14.10 | | -39.77 |
| -16.00 | -11.93 | -10.03 | -9.76 | -14.12 | | -46.51 |
| -14.00 | -12.06 | -10.14 | -9.80 | -14.29 | | -49.15 |
| -12.00 | -12.26 | -10.38 | -9.96 | -14.29 | | -43.08 |
| -10.00 | -12.49 | -10.39 | -10.19 | -14.41 | | -32.67 |
| -8.00 | -12.69 | -10.60 | -10.45 | -14.51 | | -23.61 |
| -6.00 | -12.91 | -10.84 | -10.60 | | | -17.04 |
| -4.00 | -13.09 | -10.98 | -10.68 | | | -13.77 |
| -2.00 | -13.29 | -11.32 | -10.86 | | | -11.84 |
| 0.00 | -13.53 | -11.52 | -11.10 | | | -11.16 |

TABLE 7. HEAT FLOW OF PROPYLENE GLYCOL

| Temperature °C | PG-50% (mW) | PG-55% (mW) | PG-58% (mW) | PG-60% (mW) | PG-100% (mW) |
|------------------|------------------|-------------------|--------------------|------------------|-----------------|
| -90.00 | -13.02 | -10.28 | -11.58 | -12.33 | -8.75 |
| -88.00 | -12.52 | -10.16 | -11.46 | -12.27 | -8.69 |
| -86.00 | -11.93 | -9.98 | -11.36 | -11.86 | -8.62 |
| -84.00 | -11.68 | -9.70 | -11.04 | -11.65 | -8.57 |
| -82.00 | -11.42 | -9.55 | -10.81 | -11.56 | -8.54 |
| -80.00 | -11.14 | -9.55 | -10.71 | -11.47 | -8.53 |
| -78.00 | -10.94 | - 9.35 | -10.60 | -11.57 | -8.48 |
| -76.00 | -10.84 | -9.08 | -10.62 | -11.25 | -8.45 |
| -74.00 | -10.80 | -9.13 | -10.65 | -11.21 | -8.45 |
| -72.00 | -10.51 | -9.10 | -10.67 | -11.07 | -8.47 |
| -70.00 | -10.42 | -9.14 | -10.67 | -11.10 | -8.44 |
| -68.00 | -10.41 | - 9.10 | -10.69 | -11.10 | -8.28 |
| -66.00 | -10.33 | -9.06 | -10.72 | -11.11 | -8.26 |
| -64.00 | -10.23 | -8.93 | -10.70 | -11.07 | -8.29 |
| -62.00 | -10.35 | -8.90 | -10.72 | -11.07 | -8.23 |
| -60.00 | -10.26 | -8.86 | -10.84 | -11.10 | -8.22 |
| -58.00 | -10.10 | -8.69 | -10.77 | -11.07 | -8.22 |
| -56.00 | -9.90 | -8.77 | -10.65 | -11.14 | -8.40 |
| -54.00 | -9.88 | -8.77 | -10.70 | -11.14 | -8.22 |
| -52.00 | -9.87 | -8.75 | -10.76 | -11.14 | -8.16 |
| -50.00 | -10.02 | -8.80 | -10.82 | -11.09 | -8.14 |
| -48.00 | -10.11 | -8.77 | -10.85 | -11.03 | -7.97 |
| -46.00 -44.00 | -10.41 -10.74 | -8.77 | -11.13 | -10.99 | -7.80 |
| -42.00 | -11.63 | -8.90 -8.96 | -10.95 -11.07 | -11.06 | -7.97 |
| -40.00 | -12.17 | -9.00 | -11.33 | -11.16 | -8.29 |
| -38.00 | -12.51 | - 9.10 | -11.33 | -11.24 | -8.57 |
| -36.00 | -12.67 | -9.12 | - 11.54 | -11.36 -11.34 | -8.62 |
| -34.00 | -12.58 | -8.92 | -11.54 | -11.34 | -8.66 -8.71 |
| -32.00 | -12.32 | -9.02 | -11.50 | -11.41 | -8.89 |
| -30.00 | -11.98 | -9.15 | -11.52 | -11.41 | -9.09 |
| -28.00 | -11.88 | -9.32 | -11.65 | -11.73 | -9.33 |
| -26.00 | -11.95 | -9.45 | -11.75 | -11.76 | -9.48 |
| -24.00 | -12.02 | -9.50 | -11.98 | -11.81 | -9.58 |
| -22.00 | -12.26 | -9.57 | -12.16 | -11.90 | -9.44 |
| -20.00 | -12.20 | -9.73 | -12.34 | -11.96 | -9.27 |
| -18.00 | -12.33 | -9.87 | -12.42 | -12.04 | -9.21 |
| -16.00 | -12.58 | - 9.96 | -12.37 | -12.12 | -9.35 |
| -14.00 | -12.68 | -10.19 | -12.55 | -12.24 | -9.52 |
| -12.00 | -12.91 | -10.40 | -12.80 | -12.43 | -9.57 |
| -10.00 | -13.16 | -10.60 | - 12.82 | -12.67 | -9.64 |
| -8.00 | -13.35 | -10.85 | -13.14 | -12.71 | -9.70 |
| -6.00 | -13.62 | -11.10 | -13.36 | -12.96 | -9.80 |
| -4.00 | -13.81 | -11.29 | -13.85 | -13.19 | -9.89 |
| -2.00 | -14.13 | -11.61 | -14.35 | -13.26 | -9.89 |
| 0.00 | -14.46 | -11.94 | | -13.80 | -10.04 |

TABLE 8. HEAT FLOW OF GAMMA-BUTYROLACTONE

| | (MM) | GB-100% (mW) |
|---|--|--|
| -44.00 -42.00 -40.00 -38.00 -36.00 -34.00 -32.00 -30.00 -28.00 -26.00 -24.00 -22.00 -20.00 -18.00 -16.00 -14.00 -12.00 -10.00 -8.00 -6.00 | -5.19 -5.36 -5.37 -5.88 -0.80 0.14 -3.34 -4.90 -5.87 -5.87 -5.87 -5.87 -5.98 -6.29 -6.64 -7.39 -6.64 -7.39 -6.64 -7.39 -13.54 -10.79 -10.82 -17.94 -13.54 -10.79 -10.82 -17.94 -13.54 -10.79 -10.82 -17.94 -13.54 -10.79 -10.82 -17.94 -13.54 -10.79 -10.82 -17.94 -13.54 -10.79 -10.82 -11.36 -10.79 -10.82 -11.36 -10.79 -10.82 -11.37 | (mW) -4.19 -4.19 -4.19 -4.18 -4.24 -4.30 -4.19 -4.57 -4.57 -4.57 -4.57 -4.57 -4.57 -4.57 -4.57 -5.28 -6.57 -5.88 -7.84 -15.38 -10.88 -1 |

TABLE 9. HEAT FLOW OF SALINE SOLUTION WITH 25% ETHANOL

TABLE 10. HEAT FLOW OF 1-PENTANOL 100%

TABLE 11. HEAT FLOW OF 1,2,4-TRIMETHYLBENZENZ 100%

| Temperature °C | Heat Flow (mW) |
|--|---|
| -90.00 -88.00 -86.00 -84.00 -82.00 -80.00 -78.00 -76.00 -74.00 -72.00 -70.00 -68.00 -66.00 -64.00 -62.00 -50.00 -54.00 -52.00 -50.00 -44.00 -42.00 -40.00 -38.00 -36.00 -34.00 -32.00 -30.00 -22.00 -20.00 -18.00 -16.00 -14.00 -12.00 -10.00 -18.00 -10.00 | -4.24 -4.39 -4.46 -4.48 -4.42 -4.44 -4.42 -4.60 -4.65 -4.77 -4.66 -4.79 -5.08 -5.28 -6.49 -8.08 -10.40 -15.55 -23.96 -29.10 -24.20 -15.42 -8.99 -6.26 -5.49 -5.07 -5.08 -4.80 -4.80 -4.80 -4.90 -4.81 -4.83 -4.84 -4.96 -4.99 -4.99 |

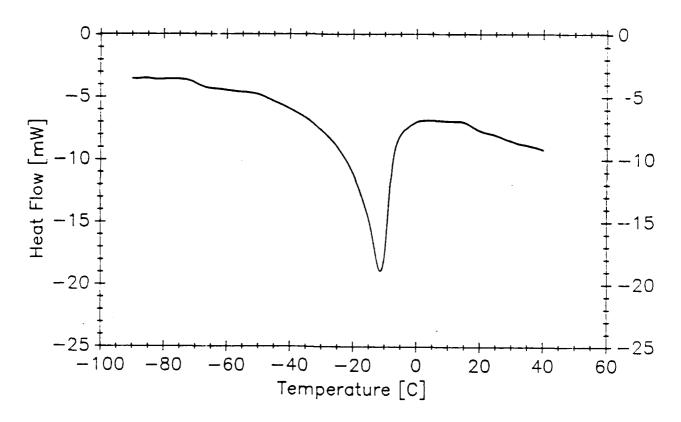


Figure 5. Heat Flow of Glycerolized RBC—A

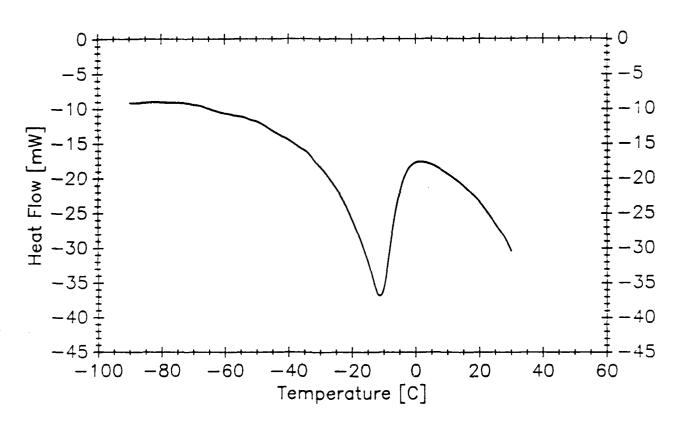


Figure 6. Heat Flow of Glycerolized RBC-B

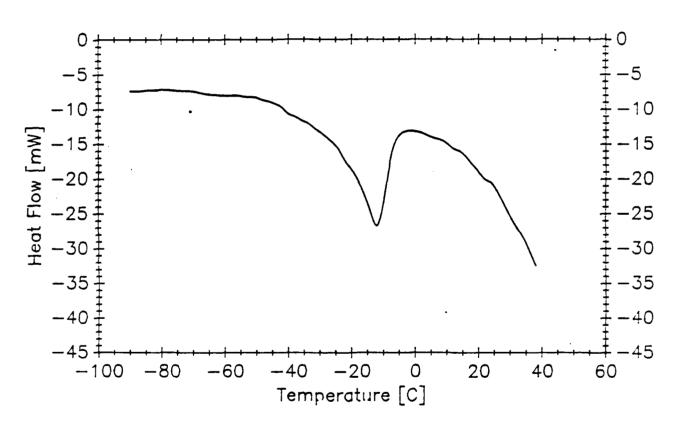


Figure 7. Heat Flow of Glycerolized RBC-C

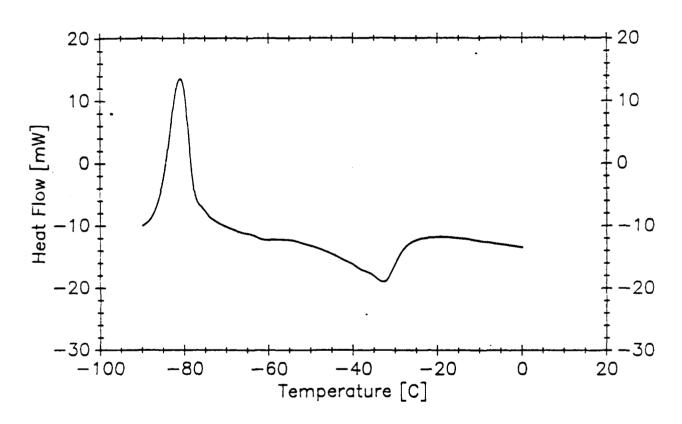


Figure 8. Heat Flow of Dowtherm SR1 50%

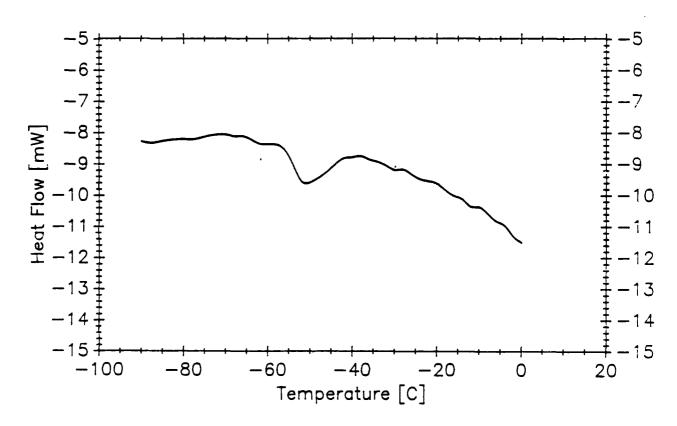


Figure 9. Heat Flow of Dowtherm SR1 52%

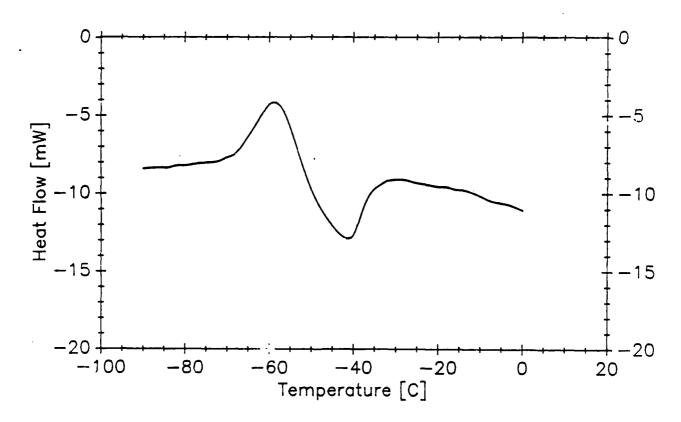


Figure 10. Heat Flow of Dowtherm SR1 55%

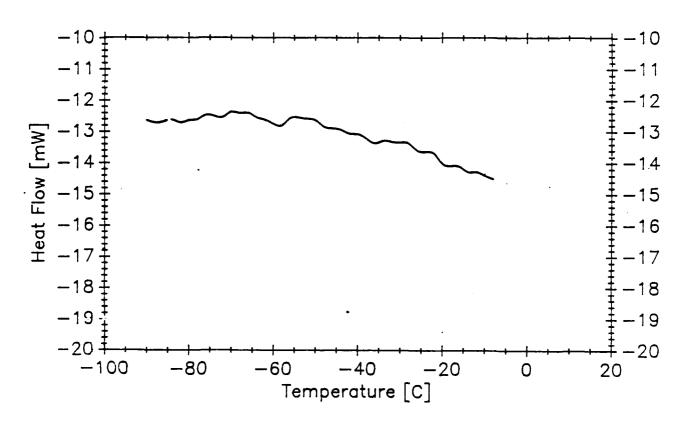


Figure 11. Heat Flow of Dowtherm SR1 58%

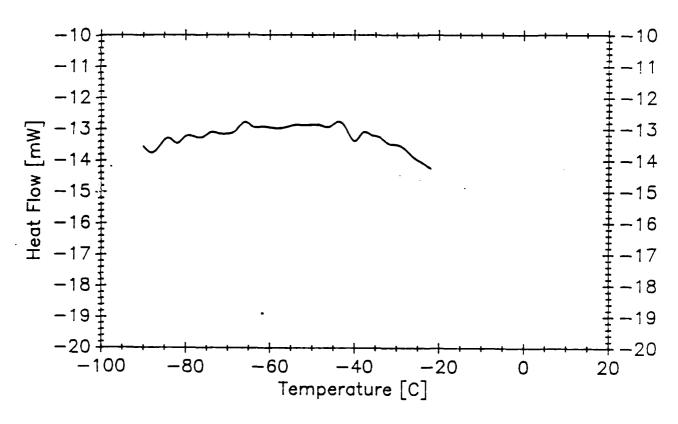


Figure 12. Heat Flow of Dowtherm SR1 60%

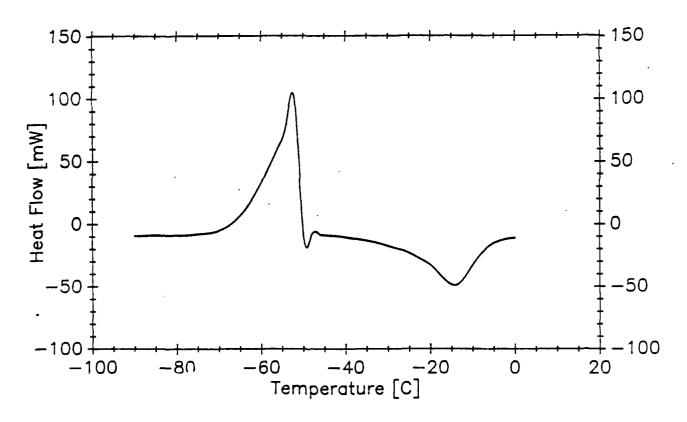


Figure 13. Heat Flow Dowtherm SR1 100%

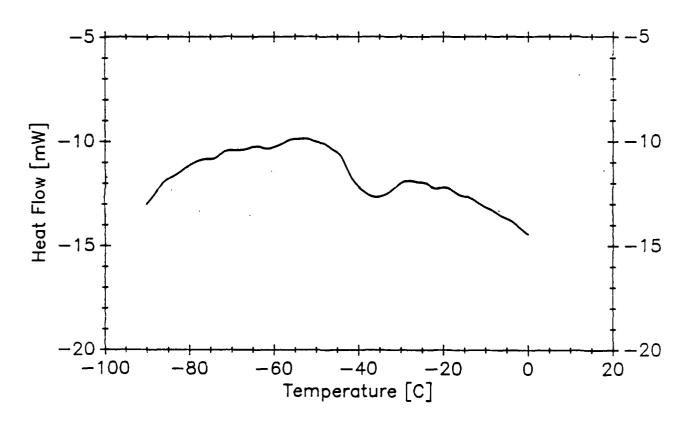


Figure 14. Heat Flow of Propylene Glycol 50%

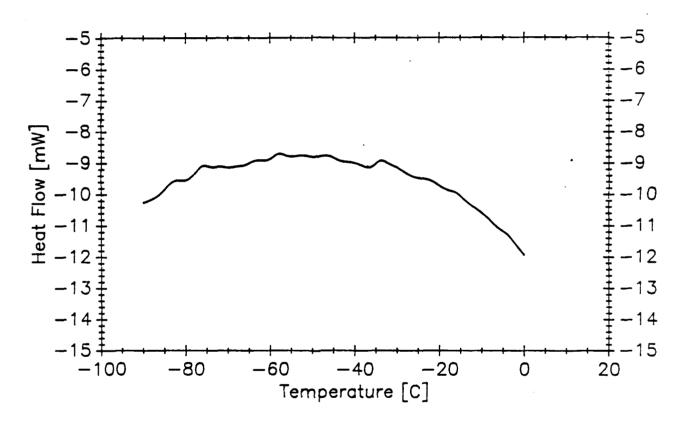


Figure 15. Heat Flow of Propylene Glycol 55%

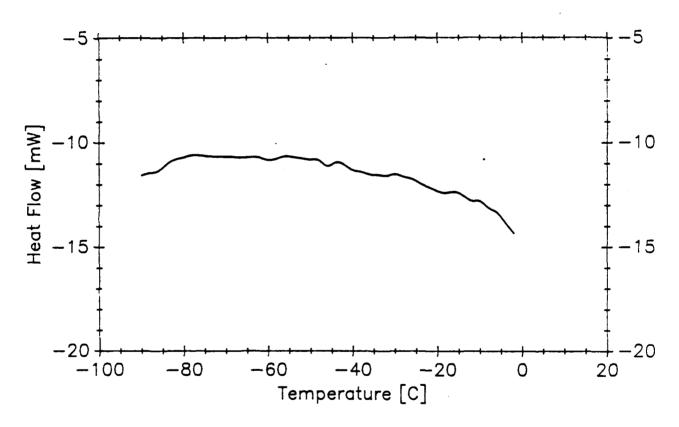


Figure 16. Heat Flow of Propylene Glycol 58%

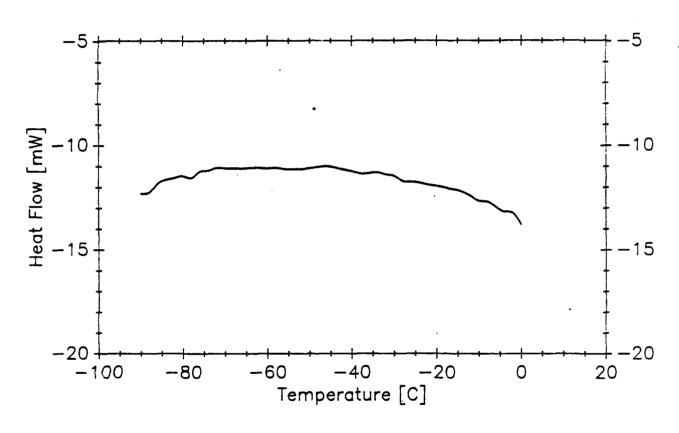


Figure 17. Heat Flow of Propylene Glycol 60%

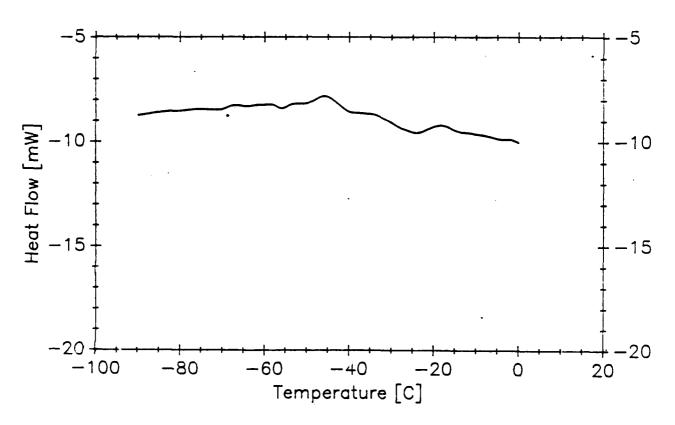


Figure 18. Heat Flow of Propylene Glycol 100%

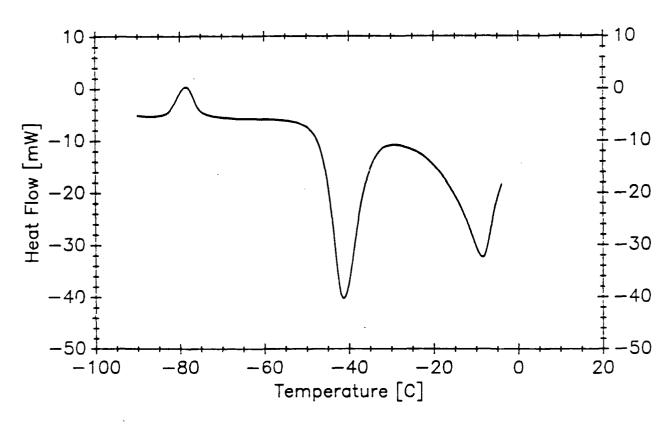


Figure 19. Heat Flow of Gamma—Butyrolactone 60%

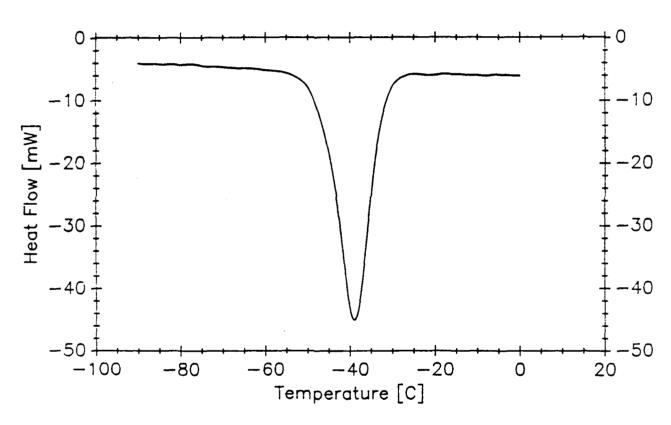


Figure 20. Heat Flow of Gamma—Butyrolactone 100%

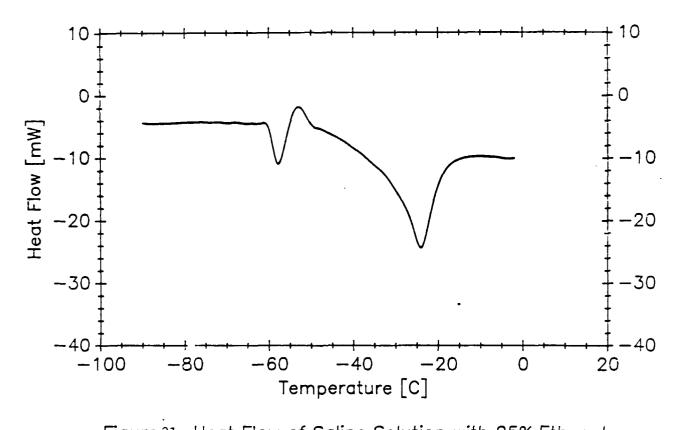


Figure 21. Heat Flow of Saline Solution with 25% Ethanol

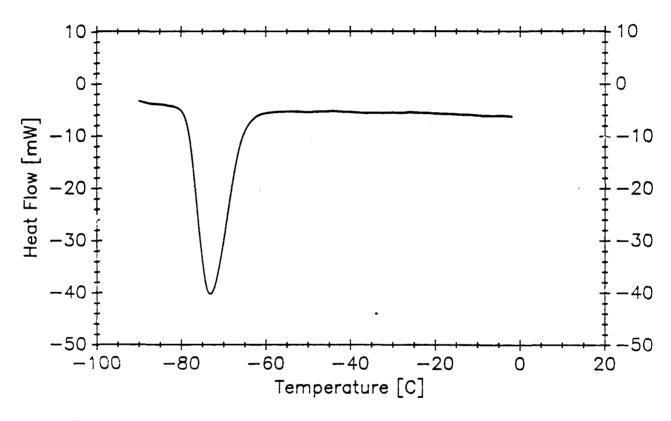


Figure 22. Heat Flow of 1-Pentanol 100%

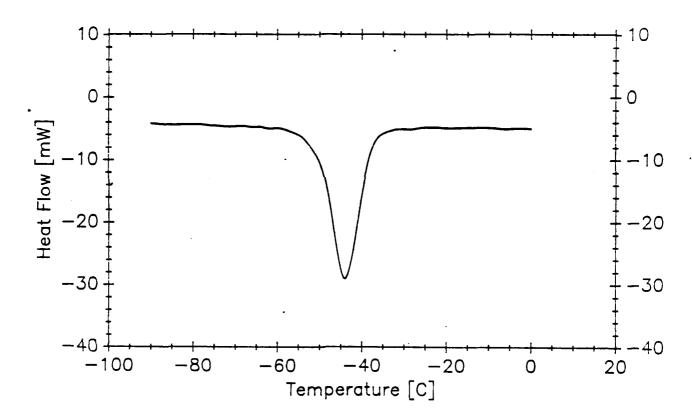


Figure 23. Heat Flow of 1,2,4—Trimethylbenzene 100%

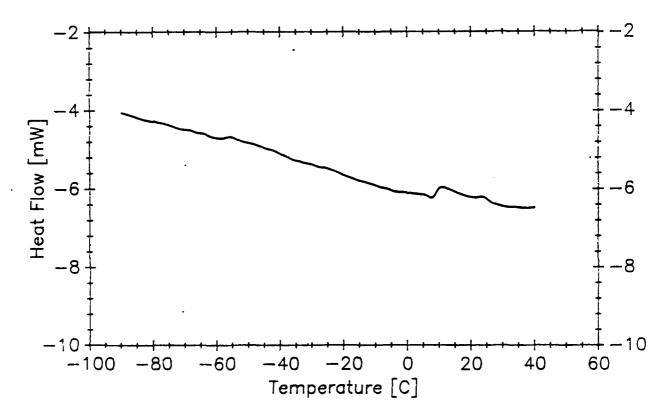


Figure 24. Heat Flow of Pan + Sapphire A

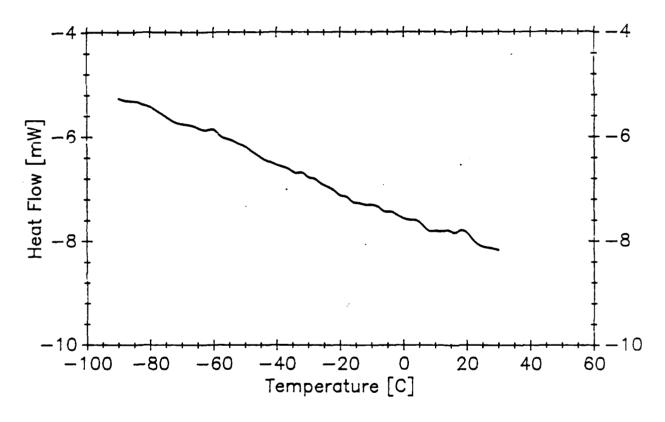


Figure 25. Heat Flow of Pan + Sapphire B

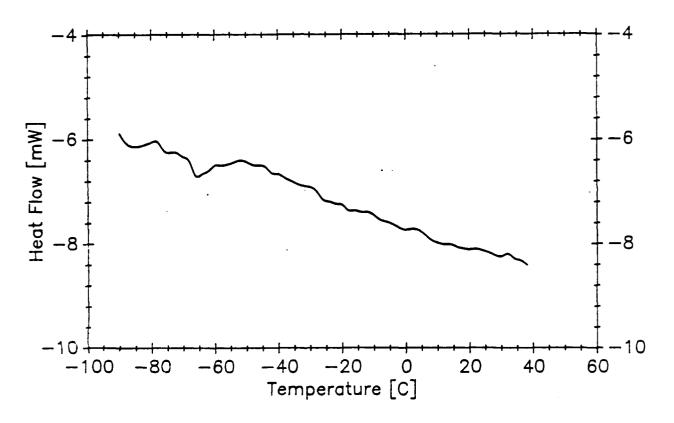


Figure 26. Heat Flow of Pan + Sapphire C

TABLE 12. SPECIFIC HEAT OF GLYCEROLIZED RED BLOOD CELL

| -90.00 |
|----------------------|
| -2.00 3.76 3.43 3.17 |

TABLE 12. (CONT.)

| Temperature | RBC-A | RBC-B | RBC-C |
|----------------|--------------|-----------|-----------|
| •C | (J/kg.°C) | (J/kg.°C) | (J/kg.°C) |
| 2.00 | 3.55 | 3.26 | 3.28 |
| 4.00 | 3.56 | 3.33 | 3.39 |
| 6.00 | 3.57 | 3.38 | 3.46 |
| 8.00 | 3.58 | 3.47 | 3.46 |
| 10.00 | 3.78 | 3.60 | 3.55 |
| 12.00 | 3.81 | 3.74 | 3.73 |
| 14.00 | 3.80 | 3.91 | 3.85 |
| 16.00 | 3.88 | 4.07 | 4.07 |
| 18.00 | 4.05 | 4.28 | 4.38 |
| 20.00 | 4.19 | 4.47 | 4.73 |
| 22.00 | 4.30 | 4.64 | 5.07 |
| 24.00 | 4.40 | 4.85 | 5.22 |
| 26.00 | 4.43 | 5.11 | 5.58 |
| 28.00 | 4.52 | 5.37 | 6.03 |
| 30.00 | 4.60 | 5.77 | 6.53 |
| 32.00 | 4.71 | | 7.04 |
| 34.00 | 4.79 | | 7.37 |
| 36.00 | 4.86 | | 7.88 |
| 38.00 40.00 | 4.95 5.08 | | 8.41 |

TABLE 13. SPECIFIC HEAT OF AQUEOUS SOLUTION OF DOWTHERM SR1

| Temperature °C | SR1-50% (J/g.°C) | SR1-52% (J/g.°C) | SR1-55% (J/g.°C) | | |
|---|--|---|--|---|--|
| -90.00 -88.00 -86.00 -84.00 -80.00 -78.00 -78.00 -74.00 -74.00 -70.00 -68.00 -64.00 -62.00 -68.00 -58.00 -58.00 -54.00 -52.00 -58.00 -54.00 -44.00 -42.00 -44.00 -42.00 -38.00 -34.00 -32.00 -38.00 -34.00 -34.00 -31.00 | 1.97 1.74 1.09 0.51 1.47 1.97 2.13 2.26 2.37 2.63 2.72 2.67 2.69 2.74 2.83 2.92 3.13 3.27 3.44 3.59 3.79 3.93 4.16 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.57 2.64 2.65 2.65 2.65 2.65 2.65 2.65 2.65 2.65 | (J/g.·C) 1.52 1.54 1.55 1.55 1.55 1.55 1.55 1.60 1.67 1.67 1.79 1.84 1.99 1.88 1.78 1.89 1.89 1.95 1.95 1.95 1.00 2.07 | 1.93 1.93 1.95 1.95 1.95 1.95 1.99 1.89 1.89 1.89 1.81 1.64 1.21 1.02 1.65 2.50 2.50 2.50 2.50 3.17 3.24 2.43 2.43 2.43 2.49 | (J/g. °C) 2.04 2.06 2.09 2.12 2.12 2.12 2.12 2.14 2.15 2.20 2.24 2.30 2.25 2.26 2.28 2.31 2.31 2.34 2.35 2.42 2.39 2.43 2.45 2.47 2.49 2.52 2.58 | 1.61 1.60 1.55 1.56 1.58 1.60 1.58 1.48 1.36 1.22 0.92 0.34 1.38 1.50 1.59 1.69 1.78 1.96 2.39 2.62 2.96 3.35 3.76 4.19 4.83 5.63 6.27 7.53 8.75 9.36 |
| -12.00 -10.00 -8.00 -6.00 -4.00 -2.00 0.00 | 2.69 2.76 2.82 2.86 2.93 2.96 3.01 | 2.13 2.15 2.20 2.25 2.29 2.36 2.40 | 2.54 2.62 2.70 2.73 2.77 2.81 2.87 | 2.58 2.62 2.66 | 8.20 6.24 4.51 3.22 2.59 2.20 2.06 |

TABLE 14. SPECIFIC HEAT OF AQUEOUS SOLUTION OF PROPYLENE GLYCOL

TABLE 15. SPECIFIC HEAT OF AQUEOUS SOLUTION OF GAMMA-BUTYROLACTONE

| | | T |
|----------------------------|--------------------|----------------------|
| Temperature • C | GB-60% (J/g.°C) | GB-100% (J/g.°C) |
| -90.00 -88.00 | 0.94 | 0.80 |
| -86.00 | 0.99 | 0.84 |
| -84.00 | 0.95 | 0.87 |
| -82.00 | 0.71 | 0.86 |
| -80.00 -78.00 -76.00 | 0.10 | 0.89 0.87 0.89 |
| -74.00 | 0.92 | 0.95 |
| -72.00 | 1.02 | 0.95 |
| -70.00 | 1.07 | 0.98 |
| -68.00 | 1.11 | 1.01 |
| -66.00 | 1.15 | 1.04 |
| -64.00 | 1.14 | 1.05 |
| -62.00 | 1.16 | 1.08 |
| -60.00 | 1.20 | 1.14 |
| -58.00 | 1.19 | 1.15 |
| -56.00 | 1.22 | 1.20 |
| -54.00 | 1.26 | 1.30 |
| -52.00 | 1.34 | 1.48 |
| -50.00 | 1.51 | 1.79 |
| -48.00 | 1.88 | 2.49 |
| -46.00 | 2.91 | 3.58 |
| -44.00 | 5.26 | 5.15 |
| -42.00 | 8.38 | 7.61 |
| -40.00 | 8.00 | 10.36 |
| -38.00 | 5.58 | 10.37 |
| -36.00 | 3.81 | 7.60 |
| -34.00 | 2.84 | 4.48 |
| -32.00 | 2.40 | 2.69 |
| -30.00 | 2.26 | 1.75 |
| -28.00 | 2.28 | 1.43 |
| -26.00 | 2.39 | 1.29 |
| -24.00 | 2.55 | 1.28 |
| -22.00 | 2.80 | 1.31 |
| -20.00 | 3.08 | 1.31 |
| -18.00 | 3.50 | 1.27 |
| -16.00 | 4.04 | 1.27 |
| -14.00 | 4.71 | 1.28 |
| -12.00 | 5.55 | 1.32 |
| -10.00 | 6.58 | 1.33 |
| -8.00 | 6.99 | 1.36 |
| -6.00 -4.00 -2.00 | 5.32 | 1.30 1.32 |
| 0.00 | | 1.34 1.35 |

TABLE 16. SPECIFIC HEAT OF SALINE SOLUTION WITH 25% ETHANOL

| Temperature | SSE |
|--|--|
| •C | (J/q.°C) |
| -90.00 -88.00 -86.00 -84.00 -82.00 -80.00 -78.00 -76.00 -74.00 -72.00 -70.00 -68.00 -66.00 -64.00 -62.00 -60.00 -54.00 -52.00 -54.00 -52.00 -54.00 -40.00 -42.00 -40.00 -38.00 -40.00 -38.00 -34.00 -32.00 -30.00 -34.00 -32.00 -30.00 -31.00 -31.00 -32.00 -31.00 | (J/g.°C) 1.47 1.52 1.52 1.52 1.59 1.49 1.49 1.47 1.54 1.51 1.60 1.58 1.63 2.02 4.18 2.63 0.72 1.70 1.96 2.22 2.48 2.80 3.58 4.09 7.80 7.80 7.97 3.80 3.77 3.80 3.87 |

TABLE 17. SPECIFIC HEAT OF 1-PENTANOL

| Temperature °C | PN-100% (J/g.°C) |
|--|--|
| | |
| -14.00 -12.00 -10.00 -8.00 -6.00 -4.00 -2.00 | 1.71 1.73 1.78 1.80 1.79 1.80 1.84 |

TABLE 18. SPECIFIC HEAT OF 1,2,4-TRIMETHYLBENZENE

| Temperature °C | TB-100% (J/g.°C) |
|---|--|
| -90.00 -88.00 -86.00 -84.00 -82.00 -80.00 -78.00 -76.00 -74.00 -72.00 -68.00 -66.00 -64.00 -62.00 -58.00 -54.00 -52.00 -50.00 -44.00 -42.00 -44.00 -32.00 -34.00 -34.00 -32.00 -34.00 -32.00 -30.00 -34.00 -32.00 -30.00 -34.00 -32.00 -30.00 | 1.10 1.16 1.19 1.21 1.20 1.22 1.21 1.30 1.35 1.35 1.44 1.52 1.68 1.92 2.43 3.17 4.79 7.45 9.06 7.54 4.75 2.71 1.84 1.59 1.46 1.47 1.38 1.35 1.47 1.48 1.35 1.47 1.47 1.38 1.35 1.47 1.47 1.38 1.35 1.47 1.47 1.38 1.36 1.35 1.47 1.47 1.47 1.47 1.47 1.47 1.48 1.47 1.47 1.47 1.48 1.49 1.49 1.49 1.49 1.49 1.49 1.49 1.49 |

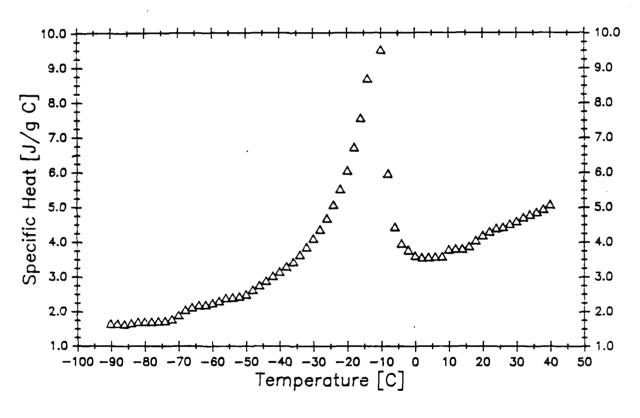


Figure 27. Specific Heat of Glycerolized RBC-A

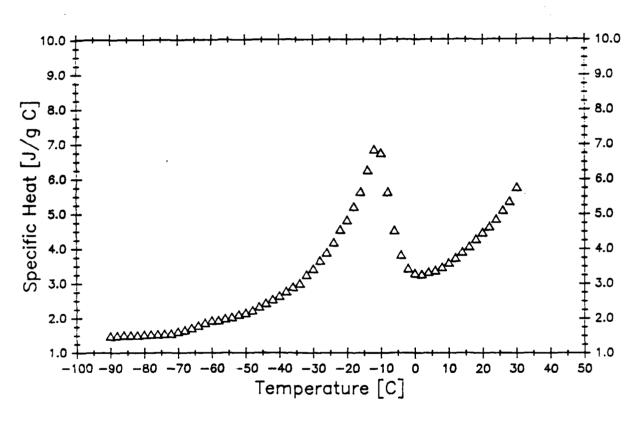


Figure 28. Specific Heat of Glycerolized RBC-B

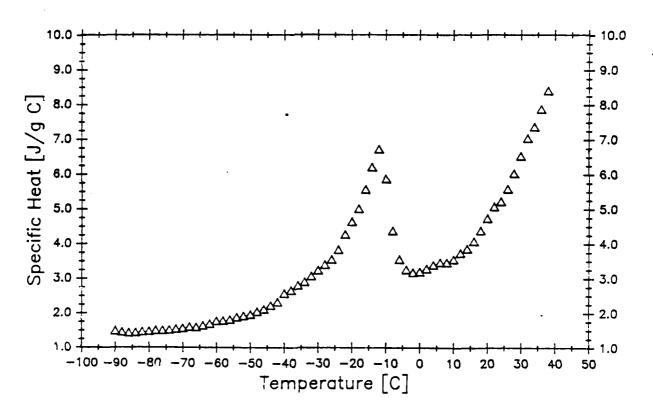


Figure 29. Specific Heat of Glycerolized RBC-C

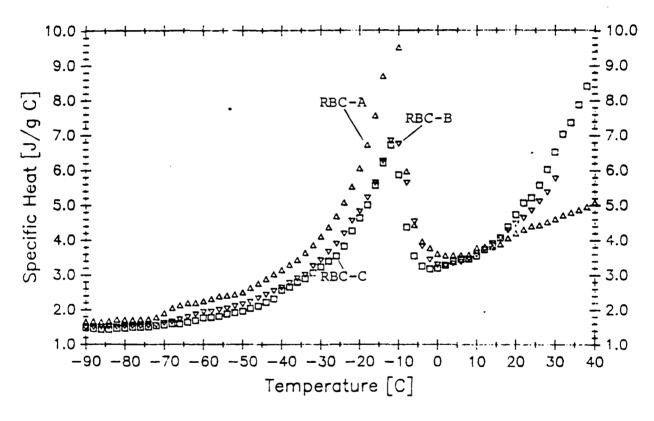


Figure 30. Specific Heat of Red Blood Cell Samples vs. Temperature

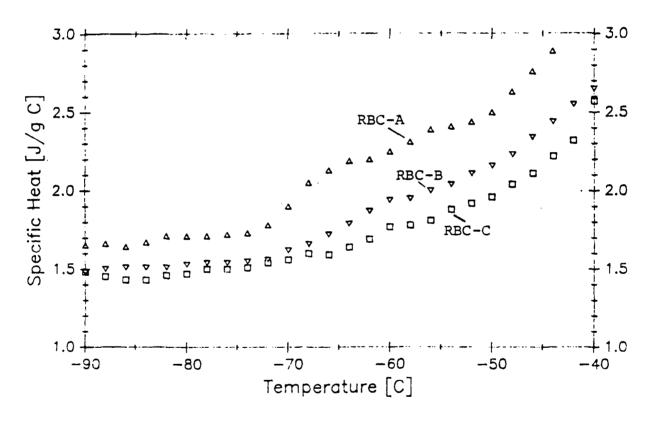


Figure 31. Specific Heat of Red Blood Cell Samples vs. Temperature

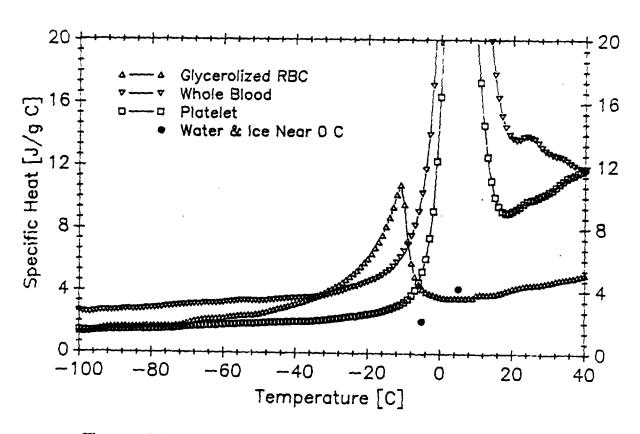


Figure 32. Specific Heat of Red Blood Cell Components

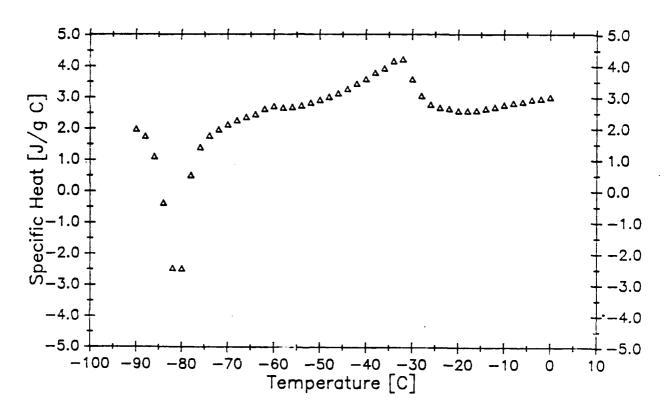


Figure 33. Specific Heat of Dowtherm SR1 50%

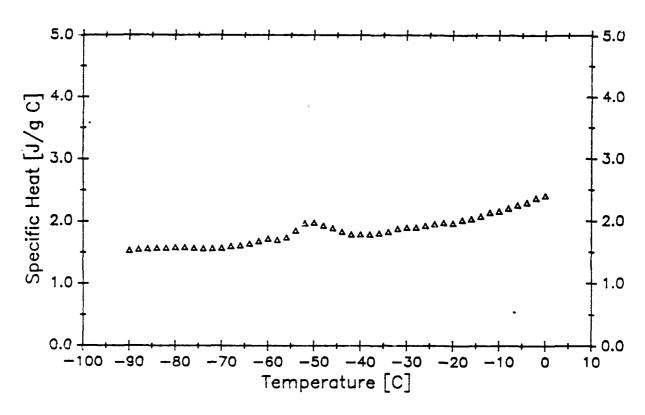


Figure 34. Specific Heat of Dowtherm SR1 52%

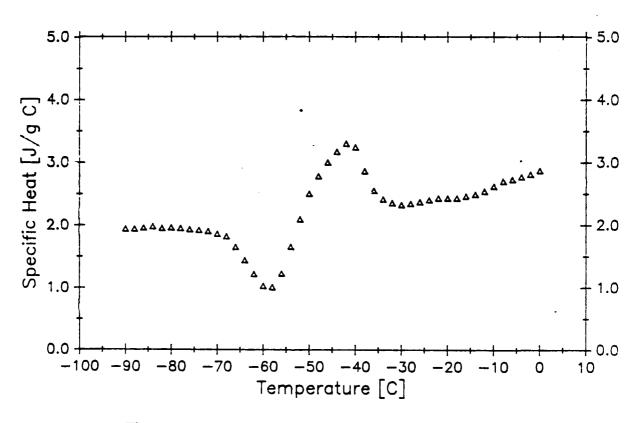


Figure 35. Specific Heat of Dowtherm SR1 55%

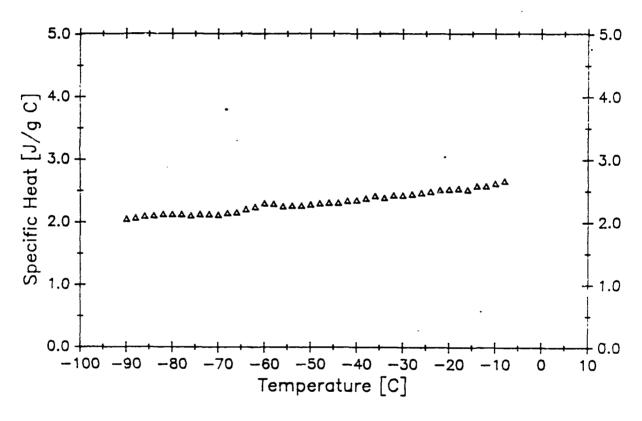


Figure 36. Specific Heat of Dowtherm SR1 58%

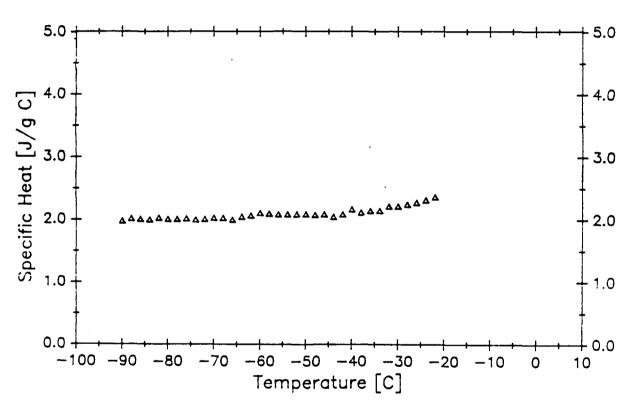


Figure 37. Specific Heat of Dowtherm SR1 60%

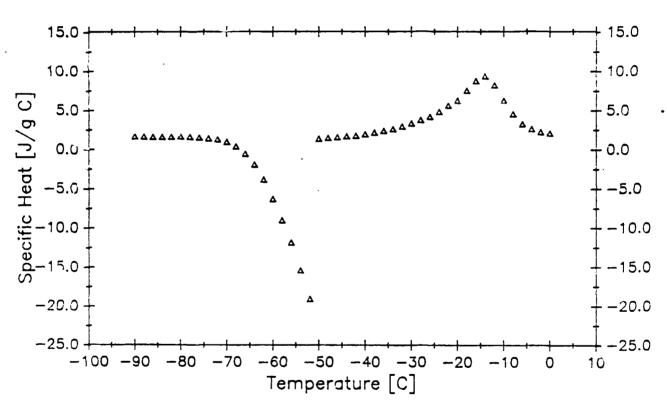


Figure 38. Specific Heat of Dowtherm SR1 100%

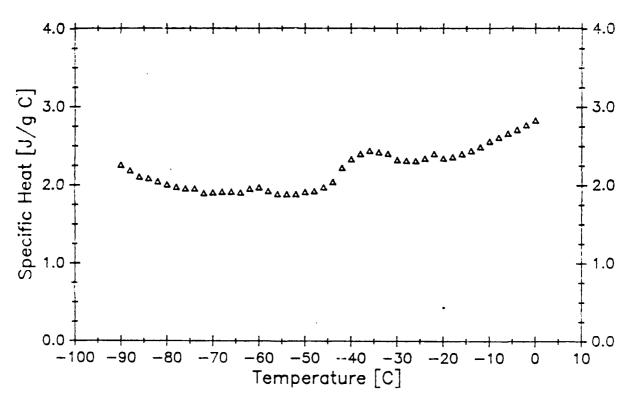


Figure 39. Specific Heat of Propylene Glycol 50%

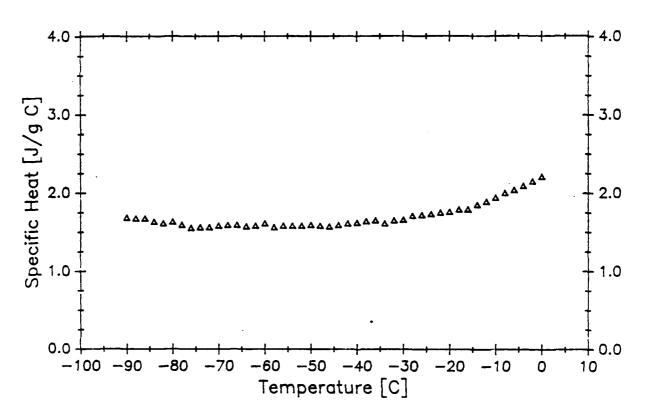


Figure 40. Specific Heat of Propylene Glycol 55%

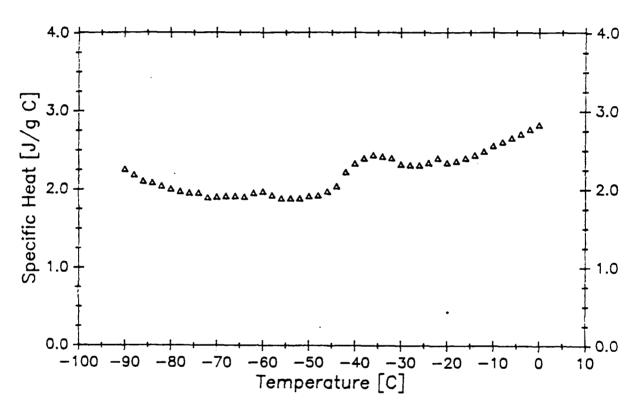


Figure 39. Specific Heat of Propylene Glycol 50%

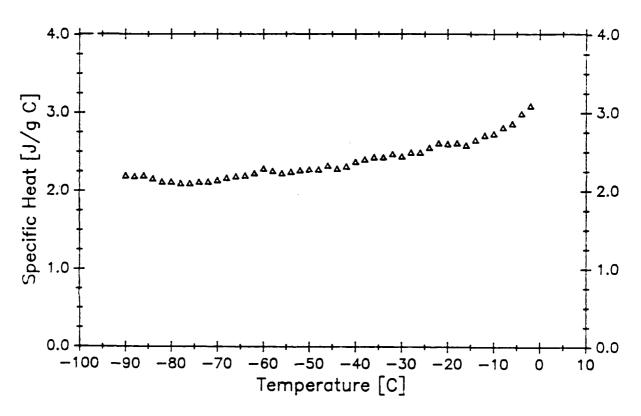


Figure 41. Specific Heat of Propylene Glycol 58%

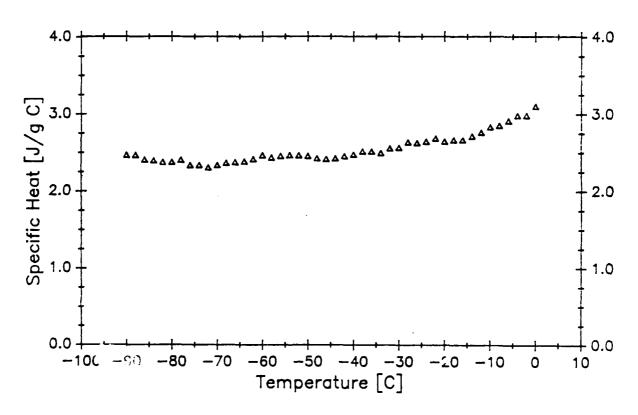


Figure 42. Specific Heat of Propylene Glycol 60%

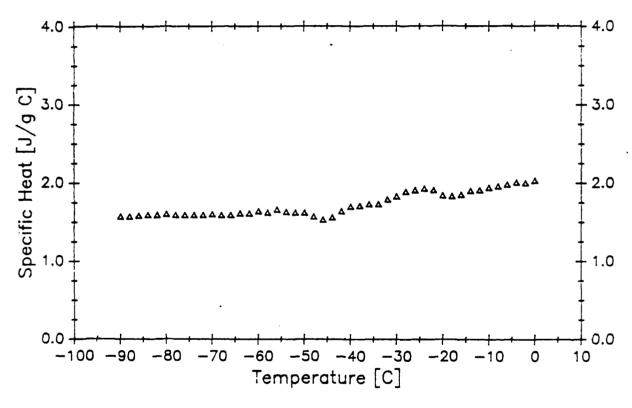


Figure 43. Specific Heat of Propylene Glycol 100%

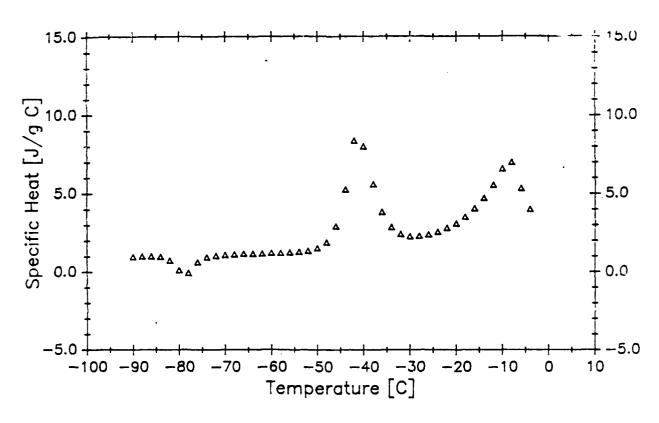


Figure 44. Specific Heat of Gamma—Butyrolactone 60%

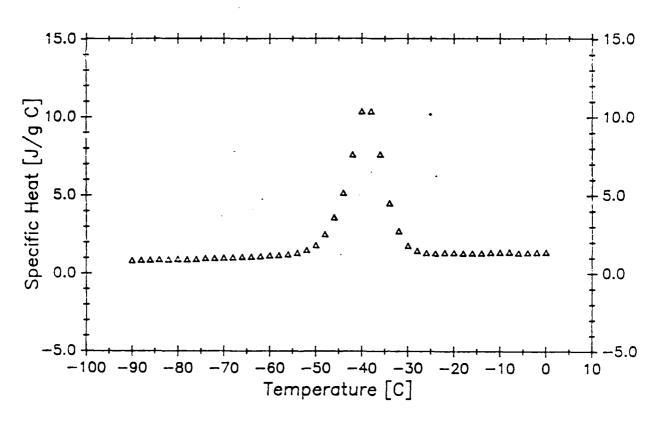


Figure 45. Specific Heat of Gamma—Butyrolactone 100%

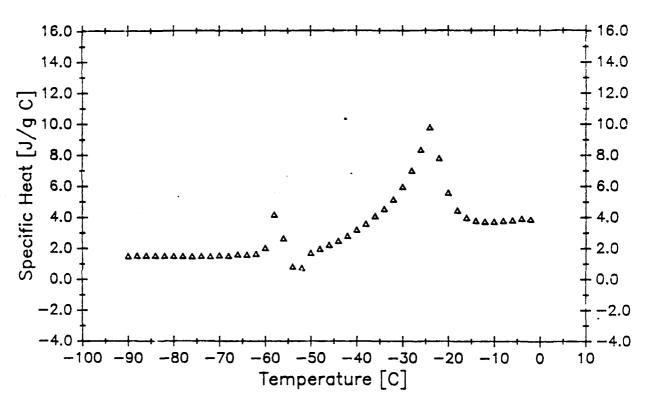


Figure 46. Specific Heat of Saline Solution With 25% Ethanol

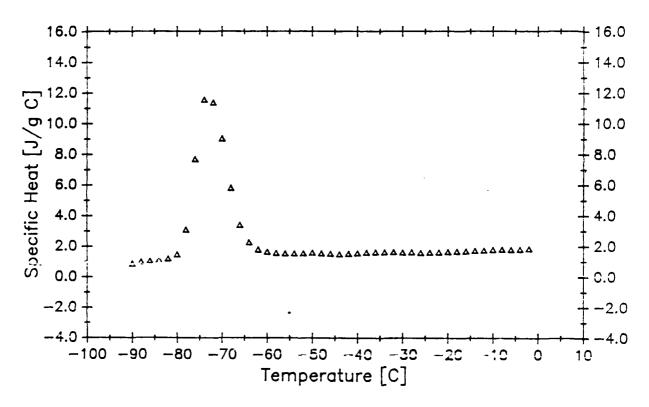


Figure 47. Specific Heat of 1-Pentanol 100%

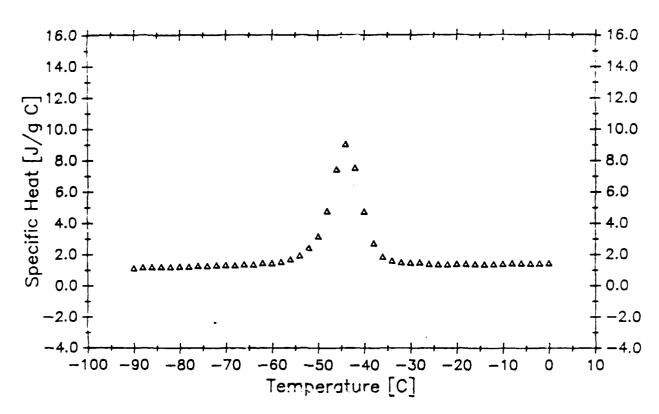


Figure 48. Specific Heat of 1,2,4—Trimethylbenzene 100%

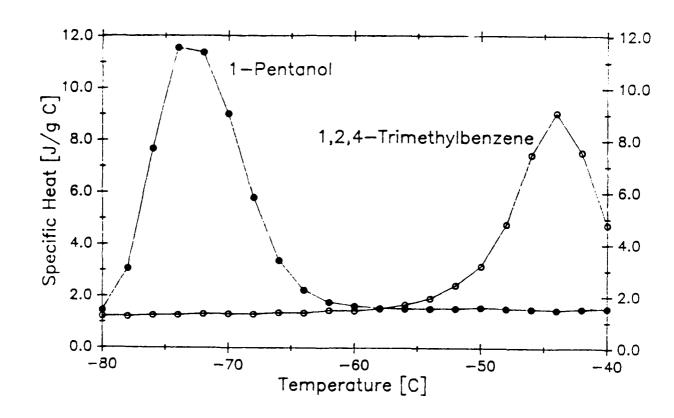


Figure 49. Specific Heat of 1—Pentanol and 1,2,4—Trimethylbenzene

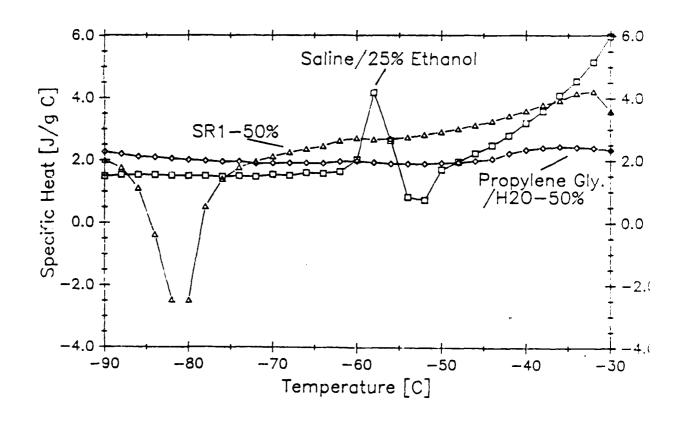


Figure 50. Specific Heat of Some Coolants vs. Temperature

TABLE 19. DENSITY OF GLYCEROLIZED RBC SAMPLES DURING FREEZING

RBC-D

| Temp(^O C) | Density | (g/ml) |
|-----------------------|---------|--------|
| 25.000 | 1.09 | 91 |
| -2.988 | 1.09 | 99 |
| -7.023 | 1.10 | 00 |
| -10.820 | 1.10 | 00 |
| -14.753 | 1.10 | 01 |
| -18.348 | 1.10 | 01 |
| -21.338 | 1.07 | 79 |
| -27.846 | 1.07 | 78 |
| -32.372 | 1.07 | 74 |
| -37.681 | 1.07 | 7 4 |
| -42.018 | 1.07 | 73 |
| -45.491 | 1.07 | 72 |
| -48.759 | 1.07 | 71 |
| -54.100 | 1.07 | 71 |

RBC-E

| Temp(OC) | Density | (g/ml) |
|----------|---------|--------|
| 25.000 | 1.09 | 2 |
| -2.925 | 1.09 | 7 |
| -6.976 | 1.09 | 9 |
| -10.794 | 1.09 | 9 |
| -14.725 | 1.10 | 0 |
| -18.287 | 1.10 | 0 |
| -21.307 | 1.07 | 79 |
| -27.810 | 1.07 | 78 |
| -32.257 | 1.07 | 72 |
| -37.556 | 1.07 | 72 |
| -41.886 | 1.07 | 71 |
| -45.352 | 1.07 | 71 |
| -48.614 | 1.06 | 59 |
| -53.945 | 1.06 | 59 |

TABLE 20. DENSITY OF GLYCEROLIZED RBC SAMPLES DURING THAWING

RBC-D

| Temp | (°C) | Density | (g/ml) |
|-----------------|------|---------|--------|
| -61 | .080 | 1.06 | 33 |
| - 55 | .930 | 1.06 | 3 |
| -43 | .312 | 1.07 | 1 |
| -41 | .844 | 1.07 | 1 |
| -40 | .273 | 1.07 | 1 |
| -33 | .315 | 1.07 | 2 |
| -25 | .283 | 1.07 | 8 |
| -16 | .531 | 1.08 | 6 |
| -7 | .560 | 1.09 | 9 |
| 1. | .905 | 1.09 | 19 |
| 11. | .478 | 1.09 | 3 |
| 20. | .726 | 1.09 | 3 |
| 25. | .000 | 1.09 | 1 |

RBC-E

| Temp | (°C) | Density | (g/ml) |
|------|-------|---------|--------|
| -61 | 1.080 | 1.06 | 55 |
| -55 | 5.930 | 1.0€ | 55 |
| -43 | 3.312 | 1.07 | 73 |
| -41 | .844 | 1.07 | '3 |
| -40 | .247 | 1.07 | 1 |
| -33 | .273 | 1.07 | 2 |
| -25 | .265 | 1.07 | 8 |
| -16 | 5.531 | 1.08 | 7 |
| -7 | .549 | 1.09 | 9 |
| 1 | .945 | 1.09 | 6 |
| 11 | .478 | 1.09 | 4 |
| 20 | .728 | 1.09 | 3 |
| 25 | .000 | 1.09 | 2 |

TABLE 21. DENSITY OF SOME COOLANTS DURING FREEZING

SR1-50%

| Temp(OC) | Density | (g/ml) |
|----------|---------|------------|
| 25.000 | 1.06 | 66 |
| -1.693 | 1.07 | ' 2 |
| -5.622 | 1.07 | 4 |
| -9.297 | 1.07 | ' 5 |
| ~13.189 | 1.07 | 8 |
| -16.699 | 1.08 | 0 |
| -20.554 | 1.08 | 0 |
| -27.064 | 1.08 | 1 |
| -31.927 | 1.08 | 3 |
| -37.339 | 1.08 | 6 |
| -41.308 | 1.08 | 0 |
| -44.686 | 1.07 | 8 |
| -47.695 | 1.07 | 3 |
| -52.905 | 1.07 | 2 |

1-Pentanol

| Temp(OC) | Density | (g/ml) |
|----------|---------|--------|
|----------|---------|--------|

| 25.000 | 0.811 | |
|----------------|-------|--|
| -3.403 | 0.821 | |
| -7.512 | 0.822 | |
| -11.576 | 0.827 | |
| -15.605 | 0.828 | |
| -19.457 | 0.832 | |
| -23.573 | 0.832 | |
| -30.637 | 0.835 | |
| -36.009 | 0.840 | |
| -41.747 | 0.841 | |
| -46.733 | 0.846 | |
| ~50.775 | 0.849 | |
| -54.666 | 0.852 | |
| -60.484 | 0.852 | |

TABLE 21. Continued

1,2,4-Trimethylbenzene

| Temp(OC) | Density (g/ml) |
|----------|----------------|
| 25.000 | 0.889 |
| -3.489 | 0.903 |
| -7.634 | 0.905 |
| -11.712 | 0.910 |
| -15.724 | 0.910 |
| -19.585 | 0.915 |
| -23.712 | 0.916 |
| -30.837 | 0.919 |
| -36.228 | 0.925 |
| -41.985 | 0.927 |
| -46.988 | 0.931 |
| -51.263 | 0.937 |
| -55.009 | 0.939 |
| -63.319 | 0.967 |

TABLE 22. DENSITY OF SOME COOLANTS DURING THAWING

SR1-50%

| Density | (g/ml) |
|---------|--|
| 1.07 | 3 |
| 1.07 | 3 |
| 1.07 | 3 |
| 1.07 | 3 |
| 1.07 | 7 |
| 1.08 | 1 |
| 1.08 | 2 |
| 1.08 | 1 |
| 1.07 | 7 |
| 1.07 | 3 |
| 1.07 | 0 |
| 1.06 | 6 |
| 1.06 | 6 |
| | 1.07 1.07 1.07 1.07 1.08 1.08 1.08 1.07 1.07 1.07 |

1-PENTANOL

| Temp (°C) | Density (g/ml) |
|---------------------|----------------|
| -64.508 | 0.863 |
| - 59.136 | 0.862 |
| - 46.715 | 0.850 |
| -44.708 | 0.849 |
| -42.805 | 0.849 |
| - 35.208 | 0.846 |
| -26.437 | 0.839 |
| -17.149 | 0.833 |
| -7.740 | 0.829 |
| 1.848 | 0.823 |
| 11.459 | 0.818 |
| 20.735 | 0.812 |
| 25.000 | 0.811 |

TABLE 22. Continued

1,2,4 Trimethylbenzene

| Temp (°C) | Density (g/ml) |
|-----------|----------------|
| -67.092 | 1.005 |
| -61.654 | 1.005 |
| -49.663 | 0.980 |
| -47.215 | 0.979 |
| -44.003 | 0.957 |
| -35.309 | 0.929 |
| -26.577 | 0.923 |
| -17.244 | 0.916 |
| -7.788 | 0.910 |
| 1.814 | 0.903 |
| 11.440 | 0.897 |
| 20.727 | 0.891 |
| 25.000 | 0.889 |

4. CONCLUSIONS AND RECOMMENDATIONS

4.1 <u>Blood</u> Some thermophysical properties of glycerolized RBCs, whole blood (CPDA-1), and platelets in the temperature range of -90 to 30 $^{\circ}$ C (-130 to 86 $^{\circ}$ F) were measured. Perhaps one of the most striking points to emerge from this investigation was the differences noticed in the properties of the 3 samples of RBC. The glycerolized RBC-C exhibited a lower heat capacity and heat of fusion than that of the other 2 samples. Table 23 presents the total amount of heat absorbed by the RBC samples in the temperature range of -80 to -40 $^{\circ}$ C (-112 to -40 $^{\circ}$ F). Note that the heat absorbing capacity of sample A is over 20% larger than that of sample C. Therefore, for safety precautions, we recommend that properties of sample C be used in the design of the shipping container.

The following properties are extracted from the tables for glycerolized RBC-C:

| Melting onset temperature | -52.0 °C (-61.6 °F) |
|---|---------------------------------|
| Melting temperature | -24.8 °C (-12.6 °F) |
| Heat of fusion | 65.2 J/g |
| Effective specific heat (-80 to -40 °C) | 1.47 to 2.57 J/g ^O C |
| Total heat capacity (-80 to -40 °C) | 71.9 J/g |

Experiments on the density of 2 samples of RBC-D and RBC-E, in the range of -61 to 25 $^{\rm O}$ C (-77.8 to 77 $^{\rm O}$ F), revealed 2.6% volumetric change. The density of the sample D increased from 1.063 g/ml at -61 $^{\rm O}$ C (-77.8 $^{\rm O}$ F) to 1.091 g/ml at room temperature.

We recommend a minimum of 3.5% extra space be provided in the shipping container for volumetric expansion of the RBCs during freezing from room temperature to -80 $^{\circ}$ C (-112 $^{\circ}$ F). This recommendation is especially significant when the blood packs are frozen with the shipping container.

Even though impressive achievements in the freezing techniques for RBCs have been realized, accurate protocols do not exist for consistent and uniform glycerolized samples of RBCs. An inspec-

TABLE 23. TOTAL HEAT ABSORBING CAPACITY OF SAMPLES

| Sample | 00 00 .0 .0 | Temp. Range -80 to -40 °C (-112 to -40 °F) J/g |
|---------------------------|----------------|---|
| Red Blood Cells: | | |
| Sample A | 94.32 | 90.90 |
| Sample B | 80.43 | 77.39 |
| Sample C | 74.85 | 71.92 |
| Ethylene Glycol (Dowtherm | SR1): | |
| 50% | 89.99 | 94.96 |
| 52% | 71.63 | 68.50 |
| 55% | 83.74 | 79.85 |
| 58% | 93.11 | 88:87 |
| 60% | 86.00 | 81.98 |
| 100% | * | * |
| Propylene Glycol: | | |
| 50% | 82.21 | 78.17 |
| 55% | 66.53 | 63.29 |
| . 58% | 92.66 | 88.43 |
| 60% | 100.82 | 96.08 |
| 100% | 67.39 | 64.2 |
| 1-Pentanol | 147.96 | 145.33 |
| Saline + 25% Ethanol | 78.28 | 75.29 |
| Gamma-Butyrolactone: | | |
| 60% | 77 . 39 | 76.58 |
| 100% | 84.42 | 82.67 |
| 1,2,4-Trimethylbenzene | 114.03 | 111.61 |

^{*} Exothermic Process

tion of the glycerolization chart in Appendix B confirms the need for a smaller, more precise range of weights for RBCs in the glycerolized solution. Note that the heat capacity experiments were conducted only on three samples of glycerolized RBCs, which may not be true representatives of the RBC population.

The unique characteristics and properties of glycerolized RBCs advises against the application of data obtained with whole blood to red cells (see Table 1), and extrapolation of experimental red cell data.

4.2 <u>Coolants</u> A number of organic compounds that appeared to be suitable for use as coolants in the shipping container were studied. Among them, the following materials stood out as the most efficient and best performed coolants for the shipping container:

1-Pentanol
1,2,4-Trimethylbenzene
Aqueous Solution Propylene Glycol 60% and 58%
Aqueous Solution of Ethylene Glycol 58% and 50%

The compounds reviewed and tested in this study had 98% or better purity. Therefore, samples from other manufacturers may have slightly different properties according to the amount and type of additives used in their solutions.

Table 23 presents the total heat absorbing capacity of the coolants. Some coolants demonstrated drastic variations in their heat capacity near -80 $^{\rm O}$ C (-112 $^{\rm O}$ F). To show this sensitivity, the total heat absorbed by the samples in the range of -82 to -40 $^{\rm O}$ C (-116 to -40 $^{\rm O}$ F) is also displayed.

Table 24 presents the cost and performance of the top coolants. Among the selected coolants, 1-Pentanol has the highest heat capacity. Even though this coolant is approved by USAF for its degree of hazard, it is a toxic, irritant, and combustible liquid. During Freeze-Thaw experiments 1-Pentanol did not exhibit any chemical instabilities.

TABLE 24. COST AND PERFORMANCE COMPARISONS OF COOLANTS

| | Total heat Capacity | Melt T | Density | Price Per Kg ^a |
|-----------------------------|--|----------|--------------------|--|
| | in the range of -80 to -40 °C (-112 to -40 °F) | | 0 25 °C (77 °F) | - |
| | J/g | °c | g/ml | \$ |
| 1-Pentanol | 145.33 | 78.5 | 0.811 | 2.16 |
| 1,2,4-Trimethyl- benzene | 111.61 | 50.3 | 0.889 | 5.00 |
| Ethylene Clycol | | | | |
| SR1-509 SR1 589 | | 48.2 | 1.063 1.074 | 0.75 ^b 0.86 ^b |
| Propylene Glycol | | | | |
| 589 609 | | <u>-</u> | 1.019 | 2.20 ^b 2.28 ^b |

a Prices are based on quantity of purchase b Prices do not include cost of preparations of acqueous solutions

⁻ No visible melt region

Trimethylbenzene demonstrated good heat absorbing capacity. It is also an irritant, combustible liquid. Its high price makes it very undesirable for use in the shipping container.

Dowtherm SR1-50% and 58% also presented good heat capacity. However, use of SR1-50% for temperatures below -80 $^{\rm O}$ C (-112 $^{\rm O}$ F) is not recommended, due to the existence of an exotherm in the range of -90 to -78 $^{\rm O}$ C (-130 to -108.4 $^{\rm O}$ F). Dowtherm SR1 solutions have, by far, the lowest price among the coolants. Nevertheless, the labor cost of preparation of the aqueous solutions of SR1 was not considered in the table of cost comparisons.

Propylene glycol 58% and 60% demonstrated good heat capacity with no visible melt region in the temperature range of experimentation. Once again, the labor cost of preparation of the aqueous solution of propylene glycol was not considered in the cost comparison.

Despite the fact that 1-Pentanol, to some degree, is toxic and has approximately 20% lower density than aqueous solutions of ethylene glycol and propylene glycol, its high heat capacity makes this coolant the best choice for the shipping container.

REFERENCES

- Valeri, C. R., Sims, K. L., Bates, J. F., Reichman, D., Lindberg, J. R., and Wilson, A. C., <u>An Integrated Liquid-</u> <u>Frozen Blood Banking System for Operational Facilities</u>, TR-82-01, Naval Blood Research Laboratory, Boston University School of Medicine, January 1982.
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- Advanced Heat Transfer, editor: Chato, B. T., University of Illinois Press, 1969.
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- 5. <u>CRC Handbook of Chemistry and Physics</u>, Weast, 64th edition, CRC Press, 1983-1984.
- 6. <u>Standards for Blood Banks and Transfusion Services</u> Tenth edition, prepared by Committee on Standards, American Association of Blood Banks, 1981.

APPENDIX A LIST OF ORGANIC COMPOUNDS

- 1 Octane
 Heat of fusion:181.57
 Melting point:-56.8
 Boiling point:125.67
- Pentanethiol
 Heat of fusion:168.2
 Melting point:-75.7
 Boiling point:126.6
- 3 cis-1-Cyano-1,3-Butadiene Heat of fusion:160.71 Melting point:-62.58 Boiling point:134.6
- Monane
 Heat of fusion:120.62
 Melting point:-53.5
 Boiling point:150.8
- 5 1-Pentanol
 Heat of fusion:111.5
 Melting point:-78.9
 Boiling point:137.3
- Propylene-Glycol(60%)
 Heat of fusion:-240
 Melting point:-58
- 7 Gamma-Butyrolactone
 Heat of fusion:111.17
 Melting point:-43.37
 Boiling point:206
- B Dimethyl formamide
 Heat of fusion:108.03
 Melting point:-60.48
 Boiling point:149.56
- 3-Methylthiophene
 Heat of fusion:107.32
 Melting point:-68.97
 Boiling point:115.44
- 4-Ethyl-1-Methylbenzene
 Heat of fusion:105.81
 Melting point:-62.35
 Boiling point:161.99
- 11 1,2,4-Trimethylbenzene
 Heat of fusion:102.68
 Melting point:-43.8
 Boiling point:169.35

- 12 1-Methyl-3-Isopropylbenzene Heat of fusion:101.92 Melting point:-63.75 Boiling point:175.14
- 13 l-cis-3-Dimethylcyclohexane
 Heat of fusion:96.43
 Melting point:-75.57
 Boiling point:120.09
- 14 Chlorobenzene
 Heat of fusion:85.35
 Melting point:-45.2
 Boiling point:132
- 15 1,3,5-Trimethylbenzene Heat of fusion:79.08 Melting point:-44.7 Boiling point:164.7
- 16 1,4-Diethylbenzene Heat of fusion:78.87 Melting point:-42.85 Boiling point:183.75
- 17 1-Methyl-2-Isopropylbenzene Heat of fusion:74.52 Melting point:-71.54 Boiling point:178.15
- 18 1-Methyl-4-Isopropylbenzene Heat of fusion:71.96 Melting point:-67.94 Boiling point:177.1
- 19 Chloropicrin
 Heat of fusion:70.21
 Melting point:-69.49
 Boiling point:111.84
- 20 Decane
 Heat of fusion:202.25
 Melting point:-29.7
 Boiling point:174.1
- 21 Undecane
 Heat of fusion:142.76
 Melting point:-25.6
 Boiling point:196
- 22 Butanol
 Heat of fusion:125.23
 Melting point:-89.8
 Boiling point:117.2

- 23 Pyrrole
 Heat of fusion:117.86
 Melting point:-23.41
 Boiling point:130-1
- Pentanoic acid or valeric acid Heat of fusion:116.44 Melting point:-33.8 to -59 Boiling point:186
- 25 1,2-Diethylbenzene
 Heat of fusion:108.49
 Melting point:-31.24
 Boiling point:183.42
- Methylchloroacetate
 Heat of fusion:104.01
 Melting point:-32.12
 Boiling point:129.82
- 27 1-trans-4-Dimethylcyclohexane
 Heat of fusion:101.61
 Melting point:-36.96
 Boiling point:119.35
- Isobutyl Cyclohexane
 Heat of fusion:100.92
 Melting point:-95
 Boiling point:171.3
- 29 2,3-Dithiabutane
 Heat of fusion:97.57
 Melting point:-84.7
 Boiling point:109.7
- 1-trans-2-Dimethylcyclohexane
 Heat of fusion:93.48
 Melting point:-88.19
 Boiling point:123.42
- Benzotrifluoride
 Heat of fusion:92.22
 Melting point:-29.11
 Boiling point:102.06
- 32 2-Ethyl-1-Methylbenzene
 Heat of fusion:88.41
 Melting point:-80.83
 Boiling point:165.15
- 1,1,2-Trichloroethane
 Heat of fusion:86.53
 Melting point:-36.6
 Boiling point:113.8

- 34 Ethylbenzene
 Heat of fusion:86.32
 Melting point:-94.98
 Boiling point:136.19
- 35 1,3-Dichlorobenzene
 Heat of fusion:85.98
 Melting point:-24.8
 Boiling point:173
- n-Butylbenzene
 Heat of fusion:83.6
 Melting point:-87.9
 Boiling point:183
- 1-cis-4-Dimethylcyclohexane
 Heat of fusion:82.93
 Melting point:-87.44
 Boiling point:124.32
- Propylcyclohexane
 Heat of fusion:82.17
 Melting point:-94.9
 Boiling point:156.7
- 39 1,3-Diethylbenzene
 Heat of fusion:81.96
 Melting point:-83.92
 Boiling point:181.1
- 40 N,N-Dibutylaniline
 Heat of fusion:79.5
 Melting point:-32.2
 Boiling point:274.75
- 41 Isopropylbenzene
 Heat of fusion:77.11
 Melting point:-96
 Boiling point:152.4
- 42 3,4-Dithiahexane
 Heat of fusion:76.15
 Melting point:-101.52
 Boiling point:153.99
- 43 Ethylcyclohexane
 Heat of fusion:74.2
 Melting point:-111.32
 Boiling point:131.78
- Tolulene or methyl benzene Heat of fusion:71.84
 Melting point:-94.99
 Boiling point:110.63

- 45 Propyl benzene
 Heat of fusion:71
 Melting point:-99.5
 Boiling point:159.2
- 46 o-Toludiene
 Heat of fusion:70.33
 Melting point:-23.68
 Boiling point:200.23

APPENDIX B GLYCEROLIZATION OF RED BLOOD CELLS FOR CRYOPROTECTION

Department of the Air Force Wilford Hall USAF Medical Center (ATC) Lackland AFB, Texas 78236-5300 Reportment of Pathology (SGHGLC8) Transfusion Branch OI 23 June 47

Adopted C/74/8 + 6 1 mo
Revieved 13 JUL 1956 7 House A

PRODUCT MANIPULATION

Glycerolization of Red Blood Cells (Muman) for Cryoprotection

(Glycerol (6.2M), Uncontrolled Freezing Rate)

- 1. PURPOSE: To identify procedures to be used in the preparation of transfusable red cells for freezing.
- 2. <u>QISCUSSION</u>: Human red blood cells collected in standard anticoequiant solutions cannot be frozen without producing hemolysis which renders the cells useless and unacceptable for transfusion. The hemolysis is ostensibly induced by osmotic stress associated with dehydration and ionic loss during freezing and thewing. It has been established by several investigators that the colligative properties of glycerol can effectively protect human red cells against freeze-them hemolysis.

In the procedure described below, a higher concentration of glycerol is used then in the rapid-freeze technique. The advantage of using a higher concentration of glycerol is that vater is bound and mitigates cellular dehydration during freezing. The increased quantity of glycerol binds more of the free vater so that less is available for participation in crystallization during freezing. In this procedure, freeze-thaw hemolysis is not a function of the freezing rate. Uncontrolled freezing rates can be used in conjunction with mechanical freezers capable of maintaining a constant temperature of -65°C or colder.

Approximately equal parts (v/v) of packed red blood cells (90 vol X) and a glycerol solution are required in this procedure. After freezing, the units are stored at a temperature of -65°C or colder. Cells cryopreserved by this method can withstand temperature variations, including thawing and refreezing (aithough not desirable), without serious increase in hemolysis.

Once frozen, the units must be stored continuously at -65°C or colder. These units can be conveniently shipped in the frozen state by packing them in dry ice (-78°C) prior to shipping. Transient variations in temperature do not significantly increase nost-they hemolysis.

In accordance with Air Force regulations, Red Blood Cells (HUMAH) Frozen may be stored for up to seven years. Antigencially rare and uncommon units have been stored in excess of seven years and transfused with satisfactory results.

J. MATERIALS:

a. Glycerol solution (57g/100ml). Each 100ml contains 57g glycerin, USP, sodium lactate, 30mg potassium chloride, USP, buffered with approximately 25mEq/l of sodium phosphate

- b. Cenister for blood freezing beg.
- c. Dielectric sealer.
- d. Blood freezing bag.
- e. Eberbach shaker. 2-speed.
- 1. Leboratory balance, 1kg capacity.
- g. Plasma expressor.
- h. 300ml Transfer Beg.
- 1. Blood product labels.
- (1) Standard full-face packed red cell label for the group/type of the unit to be frozen.
- (2) WHMC identification label. The FDA license number will be struck out with an indelible pen as frozen blood is not a licensed product at this location.
- (3) Red Blood Cells (HUHAN) Frozen add-on label placed on the full face label over the area which identifies the product type (i.e., previously Red Blood Cells (HUHAN)).
 - j. Component Source and Disposition Card (AFSC Form 3424).
 - k. 0.5ml Microtube with cap.
 - 1. 10X75mm tube.
 - a. Biological freezer capable of maintaining a temperature of -65°C or colder.

4. PROCEDURE:

- a. Preliminary steps to glycerolization:
- $% \left(1\right) \left(1\right) =0$. Allow unit to be frozen to approach room temperature for a minimum of 10 minutes.
- (2) Centrifuge the unit to obtain a 90 vol % hematocrit. Express any excess plasma from the red cell unit and retain approximately 2ml of cell free plasma in a 10X75mm tube labeled with the unit number.
- (3) Label three microtubes with the unit number and fill the tubes to within 1/4 inch of the top with cell free plasms. Cap the microtubes and set them saide to be placed in the cannister with the unit to be frozen.
- (4) Weigh the unit to be glycerolized on a belence and subtract 40g (the tare veight of the empty bag) to obtain the net veight of the packed cells. This is used to calculate the amount of glycerol required for glycerolization (step A(1)).
- (5) Close all roller clamps on the blood freezing bag and aseptically insert the cannula which has a drip chamber into the bottle of glycerol solution.
- (6) Aseptically insert the other cannuls into an used port of the unit to be frozen.
 - b. Glycerolization:

- (1) Using the packed cell weight, derived above in conjunction with the glycerolization chart (Attachment 1), determine the amount of glycerol needed for initial glycerolization and the final volume of glycerol required for cryoprotection.
- (2) Position the unit on the shaker parallel to the direction of shaker movement. Initiate shaking at the lover of the two speeds and open the roller classes leading from the glycerol bottle and to the packed cell unit. Allow the initial volume of glycerol solution to flow into the unit such that the required volume enters in no less than 3 minutes.
- (3) While shaking the unit during the initial glycerolization, switch the speed from 'low' to 'high' at least once, to assure complete mixing of the blood and glycerol. When the initial glycerol volume has been accede, close the roller clamp, turn off the shaker, and let the blood and glycerol equilibrate for 5 10 minutes.
- (4) After the equilibration period, transfer the blood and remainder of the glycerol solution, according to the glycerol chart to the freezing bag by elevating the blood collection bag and opening all roller classes on the freezing bag tubing.
- (5) Mix the cells and glycerol to achieve a homogeneous suspension. Express the excess air from the freezing bag by holding the outer port attached to the tubing upright in one head, while squeezing the bottom of the bag with the other hand, forcing the air from the freezing bag into the original blood bag. Allow a small sample of glycerolized blood to enter the trensfer tubing.
- (6) Segment the glycerolized sample of blood trapped in the transfer tubing into segments of approximately two-inch lengths, using the dielectric sealer.
 - (7) Label the unit and pilot segments prior to freezing.
- (8) Prepare a Component Source Card (frozen red cell side) by entering the following information concerning the glycerolized unit:
 - (a) Unit number.
 - (b) Collection date (of the liquid unit).
- (c) Expiration date (seven years from the date the liquid unit was collected).
 - (d) Frozen segment number.
 - (e) Unit blood group/type.
 - (f) The source of the liquid unit.
 - (g) Results of sntigen phenotyping, Direct Coombe, and CMV antibody

tests.

- (h) Under "Freezing Data"
 - 1) Date unit is prepared for freezing.
 - 2) Weight of concentrated red cells.
 - 3) Total volume of glycerol solution added.
 - 4) The manufacturer and lot number of the glycerol used.

- 5) The manufacturer and lot number of the freezing bag used.
- 6) Initials of the tech completing the glycerolisation procedure.
- (i) Autologous units should be identified conspicuously with the donors name and SSN across the bottom of the card.
- (9) Annotate the original donor card, if available, to reflect the change in statum of the red cell product from Whole Blood/Packed Red Blood Gells to Frozen Blood. Enter the date and tech'm initials in the expiration data area. This card is then treated as if the product had been transfused for accounting purposes.
 - c. Freezing and Storage
- $\ \,$ (i). Open the freezing canisters and place the freezing bag inside so that no portions of the bag are crimped.
- $% \left(2\right) =2$. Place segments and plasma microtubes at the top of the freezing bag and close the canister taking care not to cut the unit.
 - (3) Mark the end of the cannister to indicate:
 - (a) Unit number.
 - (b) Blood group/type.
 - (c) Unit expiration date.
- (4) Place the unit horizontally in a mechanical freezer until frozen (about 8 hours). Once frozen, the unit may be stored in any convenient position.
- (5) The frozen red cell card is stored in the box located in the glycerolization area with individual cards segregated by Autologous/Nomologous and blood group/type catagories.
- 5. <u>ACKNOWLEDGHENT</u>: This procedure was derived from the protocol in use at Brooke Army Medical Center, Ft Sam Houston, Texas, dated 21 October 1983.

BOBBY J. SAVYER, Hejor, USAF, BSC Chief, Trenefusion Brench

Kin D. HURPHY, Captern, USAF, HC Medical Director, Transfusion Branch

GLCEROLIZATION_CHART

| NET WEIGHT OF PACKED_CELLS | INITIAL_ADDITION | TOTAL VOLUME OF GLYCEROL_TO_BE_ADDED |
|-------------------------------|------------------|--------------------------------------|
| 75 - 125g | 15m1 | 200ml |
| 126 - 175 | 25 | 250 |
| 176 - 225 | 50 | 300 |
| 226 - 275 | 75 | 350 |
| 276 - 325 | 100 | 400 |

APPENDIX C MATERIAL SAFETY DATA SHEET FOR DOWTHERM SR-1

MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A.* Midland, MI 48674 Emergency Phone: 517-636-4400

Product Code: 25630

Page: 1

PRODUCT NAME: DOWTHERM (R) SR-1 HEAT TRANSFER FLUID

Effective Date: 02/02/89 Date Printed: 02/07/89 MSDS:000574

1. INGREDIENTS: (% w/w, unless otherwise noted)

| Ethylene glycol | CAS# 000107-21-1 | >90% |
|-----------------------|------------------|------|
| Diethylene glycol | CAS# 000111-46-6 | <5% |
| Dipotassium phosphate | CAS# 007758-11-4 | <5% |
| Water | CAS# 007732-18-5 | <5% |

This document is prepared pursuant to the OSHA Hazard Communication Standard (29 CFR 1910.1200). In addition, other substances not 'Hazardous' per this OSHA Standard may be listed. Where proprietary ingredient shows, the identity may be made available as provided in this standard.

2. PHYSICAL DATA:

BOILING POINT: 325F, 163C VAP PRESS: 2.2 mmHg @ 20C, 68F

VAP DENSITY: >1

SOL. IN WATER: Infinite.

SP. GRAVITY: 1.1295 @ 60/60F, 16C

APPEARANCE: Pink, liquid. ODOR: Pungent odor.

3. FIRE AND EXPLOSION HAZARD DATA:

FLASH POINT: 232F, 111C METHOD USED: TCC

FLAMMABLE LIMITS LFL: Not deter. UFL: Not deter.

EXTINGUISHING MEDIA: Water fog, alcohol foam, CO2, and dry chemical.

(Continued on Page 2)
(R) Indicates a Trademark of The Dow Chemical Company

* An Operating Unit of The Dow Chemical Company

Dow Chemical U.S.A.* Midland, MI 48674 Emergency Phone: 517-636-4400

Product Code: 25630

Page: 2

PRODUCT NAME: DOWTHERM (R) SR-1 HEAT TRANSFER FLUID

Effective Date: 02/02/89 Date Printed: 02/07/89

MSDS:000574

3. FIRE AND EXPLOSION HAZARD DATA: (CONTINUED)

FIRE & EXPLOSION HAZARDS: Autoignition temperature in air is 748F, 398C.

FIRE-FIGHTING EQUIPMENT: Use positive-pressure, self-contained breathing apparatus.

4. REACTIVITY DATA:

STABILITY: (CONDITIONS TO AVOID) No relevant information.

INCOMPATIBILITY: (SPECIFIC MATERIALS TO AVOID) Oxidizing material.

HAZARDOUS DECOMPOSITION PRODUCTS: None known.

HAZARDOUS POLYMERIZATION: Will not occur.

5. ENVIRONMENTAL AND DISPOSAL INFORMATION:

ACTION TO TAKE FOR SPILLS/LEAKS: Small spills: Soak up with absorbent material. Large spills: Dike and pump into suitable containers. Clean up residual with water.

DISPOSAL METHOD: Salvage, or burn in accordance with local, state, and federal regulations.

6. HEALTH HAZARD DATA:

EYE: Essentially nonirritating to eyes. Vapors or mists may irritate eyes.

SKIN CONTACT: Prolonged or repeated exposure not likely to cause significant skin irritation. May cause more severe response if skin is abraded (scratched or cut).

(Continued on Page 3)
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Product Code: 25630

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PRODUCT NAME: DOWTHERM (R) SR-1 HEAT TRANSFER FLUID

Effective Date: 02/02/89 Date Printed: 02/07/89

MSDS:000574

6. HEALTH HAZARD DATA: (CONTINUED)

reproduction in animal studies. In studies on rats, ethylene glycol has been shown not to interfere with reproduction. In studies on mice, ingestion of EG in large amounts caused a small decrease in the number of litters/pair, live pups/litter, and in live pup weight. Results of in vitro ('test tube') mutagenicity tests have been negative.

7. FIRST AID:

EYES: Irrigate immediately with water for at least 5 minutes.

SKIN: Wash off in flowing water or shower. Wash contaminated clothing before reuse.

INGESTION: If swallowed, induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person.

INHALATION: Remove to fresh air if effects occur. Consult a physician.

NOTE TO PHYSICIAN: If burn is present, treat as any thermal burn, after decontamination. Consult standard literature. Supportive care. Treatment based on judgment of the physician in response to reactions of the patient. In treatment of intoxication, the use of ethanol, hemodialysis and intravenous fluids to control acidosis should be considered.

8. HANDLING PRECAUTIONS:

EXPOSURE GUIDELINE(S): ACGIH TLV is 50 ppm ceiling for ethylene alveal.

VENTILATION: Good general ventilation should be sufficient for most conditions. Local exhaust ventilation may be necessary for some operations.

(Continued on Page 5)
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Dow Chemical U.S.A.* Midland, MI 48674 Emergency Phone: 517-636-4400

Product Code: 25630

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PRODUCT NAME: DOWTHERM (R) SR-1 HEAT TRANSFER FLUID

Effective Date: 02/02/89 Date Printed: 02/07/89

MSDS:000574

8. HANDLING PRECAUTIONS: (CONTINUED)

RESPIRATORY PROTECTION: Atmospheric levels should be maintained below the exposure guideline. When respiratory protection is required for certain operations, use an approved air-purifying respirator.

SKIN PROTECTION: Use impervious gloves when prolonged or frequently repeated contact could occur.

EYE PROTECTION: Use safety glasses. If vapor exposure causes eye irritation, use a full-face respirator.

9. ADDITIONAL INFORMATION:

REGULATORY REQUIREMENTS:

SARA HAZARD CATEGORY: This product has been reviewed according to the EPA 'Hazard Categories' promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

An immediate health hazard A delayed health hazard

SPECIAL PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE: Avoid skin and eye contact. Do not ingest.

MSDS STATUS: Revised Section 9

SARA 313 INFORMATION:

This product contains the following substances subject to the reporting requirements of section 313 of Title III of the Superfund Amendments and Reauthorization Act of 1986 and 40 CFR Part 372:

(Continued on Page 6)
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Dow Chemical U.S.A.* Midland, MI 48674 Emergency Phone: 517-636-4400

Product Code: 25630

Page: 6

PRODUCT NAME: DOWTHERM (R) SR-1 HEAT TRANSFER FLUID

Effective Date: 02/02/89 Date Printed: 02/07/89 MSDS:000574

9. ADDITIONAL INFORMATION: (CONTINUED)

CHEMICAL NAME

CAS NUMBER

CONCENTRATION

ETHYLENE GLYCOL

000107-21-1 90.4 -95.4 \$

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The Information Herein is Given in Good Faith, But No Warranty,
Express Or Implied, is Made. Consult The Dow Chemical Company
For Further Information.

^{*} An Operating Unit of The Dow Chemical Company

```
1 PROJECT : Cyrogenic System Control and Data Acquisition Software
1 PROGRAM : MEASURE.BAS (in Directory Measure)
* PROGRAMMERS : Jahan Eftekhar, Philip Wong, and David Simon
' LAST MODIFICATION : September 30, 1988
' NOTE: 1) To load Quick Basic with MC-GPIB library, use
          \qbas\Q8 /L/Q8I64.QL8
       2) To make exe copy, use
          \qbas\BC Heasure.bas/V/W;
          \qbas\LiNK Measure+Q8IB4
       3) To use MC-GPIB, merge your file with QBDECL4.BAS
       4) Before using GPIB devices, you have to initialize by
          BO% = ILFIND("DAQ")
                                           4 Assign a number to DAG
                                           ' Clear DAG
          CALL IBCLR(BD%)
       5) End of String is needed in sending GPIB command
          EOSS = CHRS(13) + CHRS(10)
       6) To select thermocouple channel, use
          CALL IBWRT(BDX,"AC19" + EOS$) ' 19 is thermal isolation block
       7) To get the reading, use
          String$ = SPACE$(13)
                                           ' Initialize a location
       CALL IBRD(BD%,A$)
                                          ' Get data in ASCII
          Reading# = VAL(A$)
                                           * Convert to DP number
      8) To control Valve (D/A V, V+, V-), use.
          CALL IBURT(BD%, "AO4,0,." + STRS(Valve) + EOSS)
            * 0 <= Velve <= 10238
           ** Valve start to open around 6000-7000
          *** Requires ramp input
      9) To control Heater (D/A H, P+, P-), use
          CALL IBWRT(BD%, "AO4,1," + STR$(Heat) + EOS$)
            * 0 <= Heat <= 10238
           ** Heat is totally off >= 5500
1 QuickBASIC Declarations Rev. C.4
' NOTE : include this file only if you are using QuickBASIC Version 4.0 or
        higher.
' Common GPIB status variables
 COMMON SHARED IBSTAX, IBERRX, IBCNTX
' GPIB Subroutine Declarations
 DECLARE SUE 183NA (BDX, BDNAMES)
 DECLARE SUB IBCAC (BDX, VX)
 DECLARE SUB IBCLR (BD%)
 DECLARE SUB IBCMD (BD%, CMD$)
 DECLARE SUB IBCHDA (BD%, CMD$)
 DECLARE SUB IBDMA (BD%, V%)
```

DECLARE SUB IBEOS (BD%, V%) DECLARE SUB IBEGT (BD%, V%) DECLARE SUB IBFIND (BONAMES, BD%) DECLARE SUB IBGTS (BD%, V%) DECLARE SUB IBIST (BDX, VX) DECLARE SUB IBLOC (BD%) DECLARE SUB IBONL (BD%, V%) DECLARE SUB IBPAD (BD%, V%) DECLARE SUB IBPCT (BD%) DECLARE SUB IBPPC (BD%, V%) DECLARE SUB IBRD (BD%, RD\$) DECLARE SUB IBRDA (80%, RD\$) DECLARE SUB IBROF (BD%, FLNAMES) DECLARE SUB IBRDI (BD%, IARR%(), CNT%) DECLARE SUB IBRDIA (BD%, IARR%(), CNT%) DECLARE SUB IBRPP (BD%, PPR%) DECLARE SUB IBRSC (BD%, V%) DECLARE SUB IBRSP (BD%, SPR%) DECLARE SUB IBRSV (80%, V%) DECLARE SUB IBSAD (BD%, V%) DECLARE SUB 18SIC (BD%) DECLARE SUB IBSRE (BD%, V%) DECLARE SUB IBSTOP (BD%) DECLARE SUB IBTMO (BD%, V%) DEÇLARE SUB IBTRAP (MASK%, mode%) DECLARE SUE IBTRG (BD%) DECLARE SUB IBWAIT (BD%, MASK%) DECLARE SUB IBWRT (BD%, WR(\$) DECLARE SUB IBWRTA (BOX, WRTS) 'DECLARE SUB IBWRTF (BDX, FLNAMES) DECLARE SUB IBWRTI (BD%, IARR%(), CNT%) DECLARE SUB IBWRTIA (BD%, IARR%(), CNT%)

' GPIB Function Declarations

DECLARE FUNCTION ILBNA% (BD%, BDNAME\$) DECLARE FUNCTION ILCACX (BDX, VX) DECLARE FUNCTION | LCLR% (BD%) DECLARE FUNCTION ILCMD% (BD%, CMDS, CNT%) DECLARE FUNCTION ILCMDAX (BDX, CMDS, CNTX) DECLARE FUNCTION ILDMAX (80%, V%) DECLARE FUNCTION ILEOSX (BD%, V%) DECLARE FUNCTION ILEOT% (BD%, V%) DECLARE FUNCTION ILFIND% (BONAMES) DECLARE FUNCTION ILGTS% (BD%, V%) DECLARE FUNCTION ILIST% (BD%, V%) DECLARE FUNCTION ILLOCX (BDX) DECLARE FUNCTION ILONE% (BD%, V%) DECLARE FUNCTION !LPAD% (80%, V%) DECLARE FUNCTION ILPCT% (BD%) DECLARE FUNCTION ILPPC% (BD%, V%) DECLARE FUNCTION | LRD% (BD%, RD%, CNT%) DECLARE FUNCTION ILRDAX (BDX, RDS, CNTX) DECLARE FUNCTION ILROF% (BD%, FLNAMES) DECLARE FUNCTION | LRD1% (BD%, | LARR%(), CNT%)

```
DECLARE FUNCTION ILRDIA% (BD%, IARR%(), CNT%)
 DECLARE FUNCTION ILRPP% (BD%, PPR%)
 DECLARE FUNCTION ILRSCX (BDX, VX)
 DECLARE FUNCTION ILRSP% (BD%, SPR%)
 DECLARE FUNCTION ILRSVX (BDX, VX)
 DECLARE FUNCTION ILSAD% (BD%, V%)
 DECLARE FUNCTION ILSICX (BDX)
 DECLARE FUNCTION ILSRE% (BD%, V%)
 DECLARE FUNCTION ILSTOP% (BD%)
 DECLARE FUNCTION ILTMOX (BDX, VX)
 DECLARE FUNCTION ILTRAP% (MASK%, mode%)
 DECLARE FUNCTION ILTRG% (BD%)
 DECLARE FUNCTION ILWAITZ (BDZ, MASKZ)
 DECLARE FUNCTION ILWRT% (80%, WRTS, CNT%)
 DECLARE FUNCTION ILWRTAX (BDX, WRTS, CNTX)
 DECLARE FUNCTION ILWRTF% (BD%, FLNAMES)
 DECLARE FUNCTION ILWRTIX (BDX, IARRX(), CNTX)
 DECLARE FUNCTION ILURTIAX (BDX, IARRX(), CNTX)
1 Program start Here
! Function : TEMPERATURE# - Return a SP number by sending a DP reading.
Subroutine : AutoControl - Automatic control to set temperature.
            : Autodisplay - Get all Thermocouple readings and display on Screen.
            : Escape · Exit program.
            : FlowMinus - Decrease nitrogen flow rate.
            : FlowPlus - Increase nitrogen flow rate.
            : GetInfo - Get temperature range of experiment and data file name.
            : HeatMinus - Decrease Heater output.
            : HeatPlus - Increase Heater output.
            : SaveMeasurements . Save all thermocouple readings in file.
            : Setup - Initialize variables, create output screen, etc
            : Shutdown - Perform system shutdown
            : TakeMeasurements . Get all thermocouple readings in an array.
                                      * Function declaration to convert thermocouple reading
                                      ' to temperature in Celsius
 DECLARE FUNCTION TEMPERATURE# (Tmp#, Ref#)
  KEY 15, CHR$(64) + CHR$(1)
                                     ! All combinations of ESC sequence
  KEY 16, CHRS(0) + CHRS(1)
  KEY 17, CHR$(32) + CHR$(1)
  KEY 18, CHR$(96) + CHR$(1)
 ON KEY(1) GOSUB ManualMode
                                     * F1 key will change to manual mode
 ON KEY(2) GOSUB SaveMeasurements
                                     ' F2 key will save data to file
                                     * F3 key will increase N2 flow
 ON KEY(3) GOSUB FlowMinus
  ON KEY(4) GOSUB FlowPlus
                                     1 F4 key will decrease N2 flow
  ON KEY(5) GOSUB HeaterMinus
                                     * F5 key will increase Heat output
                                     * F6 key will decrease Heat output
  ON KEY(6) GOSUB HeaterPlus
  ON KEY(10) GOSUB Runshell
  ON KEY(15) GOSUB Escape
                                     ' Set up ESC key for all combinations
  ON KEY(16) GOSUB Escape
  ON KEY(17) GOSUB Escape
```

ON KEY(18) GOSUB Escape

```
OPTION BASE 0
                                     Reserved for thermocouple readings (DP)
DIM D#(15)
                                     ! Reserved for Temperature readings (SP)
DIM W(15), WF(15)
CLS
PRINT
PRINT # Please turn the devices on using the following sequence : **
         1) DAG system (AUX of Power Director),"
PRINT "
         2) Circuit Power Supply (Personal Switcher),"
PRINT "
PRINT "
         3) Current Source (HP Power Supply);"
PRINT "
            On Meter Selection, both bottons should be out,"
PRINT "
             i.e. using Volt-meter and V1."
PRINT
PRINT # Warning :: Do not un-plug any device before shut down sequence is performed."
PRINT " Hit any key to continue"
LOOP UNTIL INKEYS <> ""
                                     ' Initialized DAG
BD% = [LFIND("DAQ")
CALL IBCLR(BD%)
                                      ' Ramp up valve output so that user can
FOR i = 1000 TO 10000 STEP 1000
                                      * adjust external power supply
CALL IBWRT(80%, "AO4,0,-" + STR$(i) + EOS$)
NEXT i
PRINT " On the Volt-meter of the Current Source, it should read 15V."
PRINT " Wake adjustments if necessary."
PRINT
PRINT " Hit any key to continue"
LOOP UNTIL INKEYS <> ""
                                      ! Set Valve and Heat to original starting point.
CALL IBWRT(BD%, "A04,0,-5000" + EOS$)
CALL IBWRT(BD%, "A04,1,5000" + EOS$)
GOSUB Getinfo
                                      ' Get information on the experiment
GOSUB Setup
                                      ' Initialize variables and prepare screen
                                      ! Allow keyboard interrupts
KEY(1) ON
KEY(2) ON
KEY(15) ON
KEY(16) ON
KEY(17) ON
KEY(18) ON
                                      * Do all temperature setpoint as instructed
FOR Temp = Istart TO Istop STEP Istep
    Er1 = .25
                                      ' Initially Error allowed is 0.1 (0.2 peak to peak)
    LastValve = Valve
                                      ' Modified screen records on Valve and Heat
    LastHeat = Heat
    Trial = 1
                                      ' Trial is set to 1
    Success = 0
                                      ' Set Success flag to False
```

```
* Set Condition flag to False
      Condition = 0
      CondH = 0
                                       ' Set Heating Chamber Condition flag to False
      CondC = 0
                                       * Set Cooling Chamber Condition flag to False
                                       ' Heating Chamber Temperature has a difference of 5
      TempH = Temp + 5
      adj = 200
                                        ! Initial adjustment size is 200
                                       ! Update Screen information
      LOCATE 7, 60
      PRINT ; USING "####.#"; Temp
      LOCATE 8, 60
      PRINT ; USING "####.##"; Er!
      LOCATE 9, 60
      PRINT ; USING "####"; Trial
      GOSUB AutoControl
                                        ' Automatic Control
                                        ' If everything finished, it will step into shut down
  NEXT Temp
                                       'Disable all interrupts
Shutdown:
        TIMER OFF
       KEY(1) OFF
        KEY(2) OFF
        KEY(3) OFF
        KEY(4) OFF
        KEY(5) OFF
        KEY(6) OFF
        SCREEN O
        PRINT "- SHUT DOWN PROCEDURE ACTIVATED -"
        CALL IBURT(BD%, "AO4,1,0" + EOS$)
                                              ' Turn off Heater
                                               · Close data file
        CLOSE #1
        CALL IBURT(BD%, "AO4,0,0" + EOS$)
                                               ' Turn off Valve
        PRINT
                 Please close the main valve of Nitrogen Tank."
        PRINT "--> Hit any key when ready."
        LOOP UNTIL INKEYS <> ""
        CALL IBWRT(BD%, "A04,0,-10238" + EOS$) ' Turn on Valve to release pressure
        PRINT
        PRINT "
                   Please check pressure gauge if pressure is released."
        PRINT "--> Hit any key when ready."
        00
        LOOP UNTIL INKEYS <> ""
        CALL IBWRT(BD%, "A04,0,0" + EOS$)
                                              ' Turn off valve
        PRINT
        PRINT " Please turn the devices off using the following sequence :-"
        PRINT H
                 1) Current Source (HP Power Supply),"
                 2) Circuit Power Supply (Personal Switcher),"
        PRINT " 3) DAG system (AUX of Power Director)."
        PRINT
        PRINT "- SHUT DOWN PROCEDURE FINISHED -"
        LPRINT CHR$(27); CHR$(72);
                                      ' Resets Printer to Draft Quality (IBM Proprinter)
        LPRINT CHRS(18);
                                      * Resets Printer to Standard 10 CPI (IBM Proprinter)
        WIDTH LPRINT 80
                                       ' Restores Standard 80 Character Line-Width
        END
Escape:
                                      ' Disable timer
       TIMER STOP
```

```
LOCATE 20, 40
                                      1 Confirm with user
       COLOR 14
                 Are you sure? (Y/N)"
       PRINT "
       COLOR 15
                                      ' Only accept Y or N
       DO
        ch$ = INKEY$
         ch$ = UCASES(ch$)
       LOOP UNTIL ch$ = "Y" OR ch$ = "N"
       IF ch$ = "Y" THEN
          GOTO Shutdown
                                     ' Shut down is confirmed
          ELSE
                                      ' Ignor Shut down request
               TIMER ON
               LOCATE 20, 40
               COLOR 0
               PRINT "
               COLOR 15
          END IF
       RETURN
                                      ' Get experiment bound, make decision which direction
GetInfo:
                                      1 Tstep goes
       CLS
       INPUT "Enter First Temperature point: ", Tstart
       INPUT "Enter Last Temperature point: ", Tstop
       INPUT "Enter Temperature Step: ", Tstep
       Tstep = ABS(Tstep)
       [F Tstart > Tstop THEN Tstep = -Tstep
                                      ' Get data file, open it to check if exist
GetFilename:
       INPUT "Enter Data File name: ", FileName$
       OPEN FileNameS FOR RANDOM AS #1
        IF LOF(1) <> 0 THEN
                                     ' File existed
              PRINT "File already exist! Append? Y/N ";
              PRINT
                                      ' Get Y/N response
              00
                 ch$ = INKEY$
                 ch$ = UCASES(ch$)
              LOOP UNTIL ch$ = "Y" OR ch$ = "N"
                                     ' Get another filename
               IF ch$ = "N" THEN
                      GOTO GetFilename
                                      * Check # of records
                      OPEN FileNames FOR INPUT AS #1
                      DO WHILE NOT EOF(1)
                                                      * Data is not important here
                          INPUT #1, Rec, 4(0), 4(1), 4(2), 4(3), 4(4), 4(5), 4(6), 4(7), 4(8), 4(9), 4(
                      LOOP
                                      ' Close file so it can use as append mode
                      CLOSE #1
                      OPEN Filenames FOR APPEND AS #1
                      END IF
           ELSE
                                     ' It is a new file
                CLOSE #1
                OPEN FileName$ FOR OUTPUT AS #1
                Rec = 1
        END IF
```

RETURN

```
' Adjust these parameters for program performance
Setup:
                                       ! Maximum error allowed (p to p = 1)
        CONST MaxErr = .5
                                       ' Increment of error adjustment
        CONST ErrStep = .05
                                       ! Maximum number of overshoots during oscillation cycle
        CONST MaxTrial = 12
                                       ' The time required to stay within error margin (seconds)
        CONST StableTime = 200
                                       ' The time between every screen update (seconds)
        CONST ReportTime = 10
                                       ' The rest are not adjustable
                                        * Coefficients for calculating temperature of T type
        CONST RO# = .000000525792984#
                                        ' thermocouple
        CONST R1# = .00003860071243#
        CONST R2# = 4.186486602D-08
        CONST PO# = .1238117795#
        CONST P1# = 26861.17637#
        CONST P2# = -896494.2879999999#
        CONST P3# = -46489260.88#
        CONST P4# = 12441142450#
        CONST P5# = 2275304922000#
        CONST P6# = -639949686700000#
        CONST P7# = 5.435757807D+16
        CONST P8# = -2.023615370+18
        CONST P9# = 2.8301211670+19
       EOS$ = CHR$(13) + CHR$(10)
                                       * End of Strings for GPIB-DAQ
                                       * Flag for Auto Control or Manual Mode
        Auto = 1
        Valve = 5000
                                       ' Valve initial output
        Heat = 5000
                                       ' Heat initial output
        HeatLo = Heat
                                       ' Keat minimun output
                                       ' Valve minimum output
        DALo = Valve
        SCREEN 12
                                       ' Graphics screen
        VIEW (2, 2)-(550, 25), 7
                                       ' Set graphics window for indicators
        WINDOW (5000, 1)-(10238, 100) Reference of graphics window
        LINE (5000, 40)-(10238, 60), 0, BF
                                              'Draw divider on graphics window
       IF CheckShell = 0 THEN 5
Runshell: CLS
           SHELL
           CLS
5
        LOCATE 25, 1: COLOR 12: PRINT "Printer is Off-Line!";
        WIDTH LPRINT 150
       'LPRINT CHR$(27); CHR$(71);
                                       * Sets Double-Strike NLQ Printing (IBM Proprinter)
        LPRINT CHR$(15);
                                       * Sets Condensensed Print Option (IBM Proprinter)
        LPRINT "Starting Time: "; TIMES, "Date: "; DATES, "File Name: "; FileNameS
        LPRINT : LPRINT
        LOCATE 25, 1:
                               PRINT "
                                                          .
        COLOR 2
                                       ' Label color
        LOCATE 1, 72: COLOR 9
                                       ' Make screen labels
        PRINT "Mitrogen"
        LOCATE 2, 72: COLOR 12
        PRINT "Heater"
        LOCATE 4, 1: COLOR 9
```

```
PRINT "Furnace TC Temp: "
 COLOR 12
 PRINT ""Top Reference A: "
 PRINT IN
                       8: "
 PRINT ..
                       C: "
 COLOR 4
 PRINT '"Top of Sample A: "
 PRINT "
                       B: 14
 PRIN. ..
                       c. "
 COLOR 3
 PRINT '"Btm of Sample A: "
 PRINT IN
                       B: "
 PRINT IN
                      C: "
 COLOR 11
 PRINT '"Btm Reference A: "
 PRINT III
                      B: "
 PRINT ...
                      C: "
 COLOR 7
PRINT '"Amb Temperature: "
 COLOR 6
 PRINT ""Avg Sample Temp: "
LOCATE 20, 1: COLOR 2
PRINT " Experiment Started: "
PRINT "Last Data Collected: "
PRINT "
            Current Time: "
LOCATE 5, 37
PRINT "
              Date File Name: "
LOCATE 6, 37
PRINT " File Record Number: "
LOCATE 7, 37
PRINT "Temperature Set Point: "
LOCATE 8, 37
PRINT "
           Absolute Error: "
LOCATE 9, 37
PRINT "
              Trial Number: "
LOCATE 11, 37
PRINT " TC# Currently Read: "
LOCATE 12, 37
PRINT " Reading Screened: "
LOCATE 13, 37
PRINT " File Status (1010): "
LOCATE 14, 37
PRINT "
                 TC 19 Ref#: "
LOCATE 16, 37
PRINT "
             Heater Setting: external VARIAC"
LOCATE 17, 37
PRINT "
              Valve Setting: "
COLOR 15
LOCATE 20, 22
                                       ' Output experiment start time
PRINT TIMES; " "; DATES
LOCATE 5, 60
                                       ' Output data filename
PRINT UCASES(FileNameS)
LOCATE 6, 60
                                       1 Output record number
PRINT ; USING "####"; Rec
```

```
LOCATE 23, 1

    Output function keys

        PRINT "ESC: Exit"
        PRINT " F1: Manual Control"
        RETURN
                                                 ! Get all channels thermocouple readings
TakeMeasurements:
       SumRef# = 0
        LOCATE 11, 62: PRINT USING "##"; 19;
        FOR ReadRefD# = 1 TO 25
          Readings = SPACE$(13)
                                                 I Gat Therma isolation block reading
          CALL IBWRT(8D%, "AC19" + EOS$)
          CALL IBRD(BD%, Reading$)
          SumRef# = SumRef# + VAL(Reading$)
                                                 ! This index should be different from Auto/Manual
        NEXT ReadRefD#
        Ref# = SumRef# / 25
        LOCATE 14, 61: PRINT USING "###.####"; Ref#;
        FOR Z = 0 TO 2
                                                 ' Get 12 TC readings from D/A multiplexer
          DTotal# = 0
          FOR Read0# = 1 TO 10
            CALL IBWRT(BD%, "AC" + STR$(Z) + EOS$)
50
            LOCATE 11, 62: PRINT USING "##"; Z;
            Reading = SPACES(13)
            CALL IBRD(BD%, Reading$)
            D\#(Z) = VAL(Reading$)
                                                 ' Store data in a DP array
            IF ReadTempSet = 1 THEN
              IF (Z \Leftrightarrow 0) AND (Z \Leftrightarrow 1) AND (Z \Leftrightarrow 2) AND (Z \Leftrightarrow 13) THEN
              ReadW = INT(1000 * TEMPERATURE#(D#(Z), Ref#)) / 1000
                If ReadW < (W(Z) - 1) OR ReadW > (W(Z) \neq 1) THEN
                LOCATE 12, 60: PRINT USING "####.###
                                                         "; ReadW
                GOTO 50
                END IF
             END IF
            END 1F
            LOCATE 12, 60: PRINT "
            DTotal# = DTotal# + D#(Z)
          NEXT ReadD#
          D#(Z) = DTotal# / 10: DW# = D#(Z)
        NEXT Z
        FOR Z = 0 TO 2
                                                 Convert them in temperature
            \Psi(Z) = INT(1000 + TEMPERATURE#(D#(Z), Ref#)) / 1000
                                                 ' Calculate Blood temperature
        W(14) = (W(4) + W(5) + W(6) + W(7) + W(8) + W(9)) / 6
        ReadTempSet = 1
        IF TCLabiStat = 100 THEN TCLabiStat = 0
        TCLabiStat = TCLabiStat + 1
        IF TCLabiStat = 1 THEN
          LPRINT
          LPRINT " N2
                             TR 1
                                     TR 2
                                              TR 3
                                                      TS 4
                                                              TS 5
                                                                       TS 6 ";
          LPRINT " BS 7
                             BS 8
                                      BS 9
                                              BR 10 BR 11 BR 12
```

Amb

Avg

Time"

```
LPRINT
       END 15
       LPrintStat + LPrintStat + 1
       IF LPrintStat = 30 THEN
        LPrintStat = 0
         FOR ZValue = 0 TO 2
        'UF(2Value) + ((U(2Value) + 40) * (# / 5)) - 40
         LPRINT USING "####.###"; W(ZVelue);
         IF ZValue = 0 THEN LPRINT = "; TIMES
         MEXT 2Value
       END 15
       CheckBood # CheckBoad + 1
       IF CheckRead = 11 THEM CheckRead = 1
       LOCATE 13, 62: PRINT USING "00"; CheckRead;
       LOCATE 17, 59: PRINT Valve; = ";
       IF CheckRead = 10 THEN
         WRITE 81, Rec. W(0), W(1), W(2), W(3), W(4), W(5), W(6), W(7), W(8), W(9), W(10), W(11), W(12), W(
         Rec = Rec = 1
       LOCATE 6, 60: PRINT USING "####"; Rec;
       RETURN
                                               · Store data into file
SaveMessurements:
       IF Auto . 1 THEM
                                               " Meed to know where It was in Auto or Manual
          KEY(1) OH
          TIMER OFF
          ELSE
             KEY(3) STOP
              KEY(4) STOP
              KEY(5) STOP
              KEY(6) STOP
       END IF
       GOSUB Takemessurements
                                               ' Take all channels readings them store them
        WRITE #1, Rec, W(D), W(T), W(Z), W(S), W(6), W(5), W(6), W(7), W(8), W(9), W(10), W(11), W(12), W(13
       'Rec + Rec + 1
                                               ' Update record number and time
       LOCATE 6, 60
        PRINT USING "####"; Rec
       LOCATE 21, 22
       PRINT TIMES
       CôndC = 0
        Condit = 0
        Success = 1
                                               * Turn on Success Flag so it will go for next temperature se
                                               * Return using the info of whether
        IF Auto . 1 THEN
          TIMER ON
                                               1 it was in Auto or Manual
           KEY(1) ON
          FLSE
              KEY(3) ON
              KEY(4) ON
              KET(5) ON
              KEY(6) ON
        END IF
        RETURN
```

```
AutoControl:
                                              ' Auto control Mode
        ON TIMER(ReportTime) GOSUB Autodisplay | Update Screen in constant interval
        TIMER ON
        WHILE Success = 0
                                              1 Turn off Success flag
        ConRds = SPACES(13)
                                              ' Get thermo isolation block reading
        CALL IBWRT(BD%, "AC19" + EOSS)
        CALL IBRD(BD%, ConRd$)
        Rf# = VAL(ConRdS)
        TC1T = 0
        FOR ReadTC1 = 1 TO 20
        10
        ConRdS = SPACES(13)
        CALL IBRD(BD%, ConRd$)
        TC1 = TEMPERATUPE#(VAL(ConRd$), Rf#)
        IF ReadTempSet = 1 THEN
         IF TC1 < (TC1A - 1) OR TC1 > (TC1A + 1) THEN 10
        END IF
        test21 = TC1 - Temp
                                            ' Check Cooling Chamber
        IF test21 < Q AND Valve - adj >= DALO THEN
        Valve = Valve - adj | too cold
       ELSEIF test2! > 0 AND Valve + adj <= 10238 THEN
       Valve = Valve + adj' increase flow
       TC17 = TC1T + TC1
       NEXT ReadTC1
       TC1A = TC1T / 20: TC1 = TC1A
       TC4T = 0
       FOR ReadTC4 = 1 TO 20
20
       CALL IBWRT(BD%, "AC4" + EOS$)
       ConRdS = SPACES(13)
       CALL IBRD(BD%, ConRd$)
                                           ' Get TC4-Heating Chamber reading
       TC4 = TEMPERATURE#(VAL(ConRd$), Rf#)
       IF ReadTempSet = 1 THEN
        IF TC4 < (TC4A - 1) OR TC4 > (TC4A + 1) THEN 20
       END IF
      'KEY(1) STOP
                                            ' Hold F1 key
      Test11 = TC4 - TempH
                                           ' Check Heating Chamber
      IF Test11 > 0 AND Heat - adj >\approx HeatLo THEN . When too hot
        Heat = Heat - adj
                                                    ' Heat is reduced
      ELSEIF Test11 < 0 AND Heat + adj <= 10238 THEN ' When too cool
      Heat = Heat + adi
                                                   ' Heat is increased
      END JF
      TC4T = TC4 + TC4T
      NEXT ReadTC4
      TC4A = TC4T / 20: TC4 = T.4A
```

```
· Hold F1 key
KEY(1) STOP
                                       ' Check Heating Chamber
'Test1| = TC4 - TempH
                                               ' When too hot
'IF Test1! > 0 AND Heat - adj >= HeatLo THEN
                                               ! Heat is reduced
· Heat = Heat - adj
'ELSEIF Testii < 0 AND Heat + adj <= 10238 THEN ' When too cool
                                              ' Heat is increased
'Heat = Heat + adj
'END IF
CheckPrint = CheckPrint + 1
LOCATE 25, 60. PRINT CheckPrint;
IF CheckPrint = 100 THEN
  LPRINT USING "####.###"; W(0); TC1; W(2); W(3); TC4; W(5); W(6); W(7)
END IF
IF CheckPrint = 101 THEN CheckPrint = 0
CALL IBURT(BD%, "AO4,1," + STR$(Heat) + EOS$)
                                                       ' Update index
IF Heat > LastHeat THEN LINE (LastHeat, 20)-(Heat, 20), 4 ELSE LINE (LastHeat, 20)-(Heat, 20), 7
                                                       ' For index purpose
LastHeat = Heat
 * * Next line is suspended for testing:
   IF (CondH = 1 AND Test1! <= 0) THEN HeatLo = Heat 'Register Min Heat needed
 IF ABS(Test1!) <= Er! THEN CondH = 1 ELSE CondH = 0 ' If within margin, turn-on CondH
                                        ' Check Cooling Chamber
test2! = TC1 - Temp
'IF test2! < J AND Valve - adj >= DALO THEN
'Vaive = Valve - adj ' too cold
'ELSEIF test2! > 0 AND Valve + adj <= 10238 THEN
'Valve = Valve + adj' Increase flow
 CALL IBURT(BD%, "AO4,0,-" + STR$(Valve) + EOS$)
                                                      ' Update index
 IF Valve > LastValve THEN LINE (LastValve, 83)-(Valve, 83), 1 ELSE LINE (LastValve, 83)-(Valve, 83
                                                        ' For index purpose
 LastValve = Valve
 KEY(1) ON
                                                        ' Enable F1 interrupt
 ' * Next line was suspended for testing
 ' IF (CondC = 1 AND test2! >= 0) THEN DALO = Valve
                                                         * Register Min Flow needed
 IF ABS(test2!) <= Er! THEN CondC = 1 ELSE CondC = 0 'Turn on CondC if within margin
 I Condition = 0 THEN
                                        1 It was not in margin before
    IF CondH = 1 AND CondC = 1 THEN
                                        ' but it is in margin now
                                        ! Change timer to wait for settling time
       ON TIMER(StableTime) GOSUB SaveMeasurements
      TIMER ON
      LOCATE 24, 40
      COLOR 14
       PRINT "Critical period. Please do not disturb!"
       COLOR 15
       Condition = 1
                                        ' Turn on overall condition
                                        ' It was in margin before
    ELSEIF CondH <> 1 OR CondC <> 1 THEN
```

```
Condition = 0
                                             ' Change timer back to report purpose
                 TIMER OFF
                 ON TIMER(ReportTime) GOSUB Autodisplay
                 LOCATE 24, 40
                 PRINT "
                                             1 It is a overshoot, update Trial and Err
                 Er! = Er! + ErrStep
                                                            · still allow
                       Trial = 1
                       adj = 200
                       LOCATE 8, 60
                       PRINT : USING "####.##": Er!
                   ELSE
                       Trial = Trial + 1
                                            Next Trial number, update Adj
                       adj = INT(200 / Trial)
                END IF
                LOCATE 9, 60
                                             ' Update Screen
                PRINT ; USING "####"; Trial
       END IF
       LOCATE 5, 18
       PRINT USING "####.###"; TC1
       LOCATE 8, 18
       PRINT USING "####.###"; TC4
       LOCATE 22, 22
       PRINT TIMES
       WEND
       RETURN
Autodisplay:
                                            ' Put data from array to screen
         GOSUB TakeMeasurements
         FOR Z = 0 TO 2
             LOCATE 2 + 4, 18
             WF(Z) = ((W(Z) + 40) + (9 / 5)) - 40
             PRINT USING "####.### "; WF(Z);
             IF Z < 10 THEN
             "LOCATE Z + 4, 30: PRINT USING "##"; Z;
             IF Z >= 10 AND Z <> 14 THEN
              LOCATE Z + 4, 31: PRINT USING "##"; Z:
             END IF
             IF Z <> 14 THEN
              LOCATE Z + 4, 27: PRINT "(TC";
             END IF
             IF Z < 10 THEN
              LOCATE Z + 4, 32: PRINT ")";
             END IF
             IF Z >= 10 AND Z <> 14 THEN
              LOCATE Z + 4, 33: PRINT ")";
             END IF
             IF Z = 14 THEN
              LOCATE Z + 4, 27: PRINT "(TC 40-TC 9)";
             END IF
             NEXT Z
         RETURN
```

```
* Manual control mode
ManualMode:
                                               ! Modify Auto control Flag
       Auto = 0
       KEY(1) OFF
                                                ' Disable interrupts
       TIMER OFF
       KEY(3) ON
       KEY(4) ON
       KEY(5) ON
       KEY(6) ON
       KEY(10) ON
       Valve = 5000
                                               * Update function key manual
       LOCATE 23, 1
       PRINT "ESC: Exit"
       PRINT " F1: Auto Control "
       PRINT # F2: Save Data to File #
       PRINT " F3: Decrease Nitrogen Flow Rate"
       PRINT # F4: Increase Nitrogen Flow Rate"
       PRINT " F5: Decrease Heater Output"
       PRINT " F6: Increase Heater Output";
       DO
                                                ' Update readings
         KEY(2) STOP
         GOSUB Autogisplay
         LOCATE 22, 22
         PRINT TIMES
         KEY(2) ON
                                                ' Check if F1 is pressed
      LOOP UNTIL INKEYS = CHR$(0) + CHR$(59)
       LastValve = Valve
                                                ' Update Valve and Heat value
       LastHeat = Heat
       Trial = 1
                                                * Restart Trial = 1
       CondH = 0
                                                ! All conditions false
       CondC = 0
       Condition = 0
       LOCATE 9, 60
                                               ' Update screen
       PRINT ; USING "####"; Trial
       LOCATE 24, 1
       PRINT " F1: Manual Control"
        PRINT "
        PRINT "
        PRINT "
        PRINT "
        PRINT "
        Auto = 1
                                               ' Turn on Auto Flag
        KEY(3) OFF
                                               ' Get ready to Auto Control
        KEY(4) OFF
        KEY(5) OFF
        KEY(6) OFF
        TIMER ON
        KEY(1) ON
        RETURN
FlowPlus:
                                                ' Increase Valve output in Manual mode
        IF Valve <= 10238 THEN
           Valve = Valve + 25: IF Valve > 10238 THEN Valve = 10238
           LINE (Valve, 83) (Valve - 25, 83), 1
                                                     ' Update index
           CALL IBWRT(BD%, "AO4,0,." + STR$(Valve) + EOS$)
           LOCATE 17, 59: PRINT Valve; " ";
```

```
RETURN
FlowMinus:
                                               ! Decrease Valve output in Manual Mode
       IF Valve >= 5000 THEN
          IF Valve = 10238 THEN Valve = 10250
          Valve = Valve - 25
          LINE (Valve, 83)-(Valve + 25, 83), 7
                                                     ' Update index
          IF Valve < DALO THEN DALO = Valve
          CALL IBURT(SDX, "AO4,0,-" + STRS(Valve) + EOSS)
          LOCATE 17, 59: PRINT Valve; " ";
        END IF
        RETURN
HeaterPlus:
                                               ' Increase Heat output in Manual mode
        IF Heat <= 10038 THEN
          Heat = Heat + 200
          LINE (Heat, 20)-(Heat - 200, 20), 4 ' Update index
          CALL IBWRT(BD%, "AO4,1," + STR$(Heat) + EOS$)
        END IF
       RETURN
HeaterMinus:
                                               ' Decrease Heat output in Manual mode
       IF Heat >= 5200 THEN
          Heat = Heat - 200
       LINE (Heat, 20)-(Heat + 200, 20), 7 1 Update index
          IF Heat < HeatLo THEN HeatLo * Heat
          CALL IBWRT(BD%, "AO4,1," + STR$(Heat) + EOS$)
       END IF
       RETURN
                                               * Convert thermocouple reading to temerature
FUNCTION TEMPERATURE# (Tmp#, Ref#)
    T# = Ref# * 10
    V# = R0# + T# * (R1# + T# * R2#) + Tmp#
   A1# = P5# + V# * (P6# + V# * (P7# + V# * (P8# + V# * P9#)))
```

TEMPERATURE# = PG# + V# * (P1# + V# * (P2# + V# * (P3# + V# * (P4# + V# * A1#))))

END IF

END FUNCTION